Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

The impact of interplay between electronic and steric effects on the synthesis and the linear and non-linear optical properties of diketopyrrolopyrroles bearing benzofuran moieties

Supporting Information

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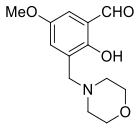
General information

All chemicals were used as received unless otherwise noted. Reagent grade solvents (MeCN, CH_2Cl_2, hexane, toluene) were distilled prior to use. All reported NMR spectra were recorded on 500 MHz spectrometer unless otherwise noted. Chemical shifts (δ ppm) for ^{1}H and ^{13}C NMR were determined with TMS as the internal reference. UV-Vis absorption spectra were recorded in toluene. Chromatography was performed on silica (Kieselgel 60, 200-400 mesh). Mass spectra were obtained with EI ion source and the EBE double focusing geometry mass analyzer or spectrometer equipped with electrospray ion source with q-TOF type mass analyzer. Compounds **1a**-**1d** and **1f**-**1l** are commercially available.

Synthesis

2-Hydroxy-5-methoxy-3-(morpholinomethyl)benzaldehyde (1e)

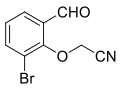


Morpholine (260 μl, 3.0 mmol), paraformaldehyde (90 mg, 3.0 mmol) and 2-hydroxy-5-methoxybenzaldehyde (380 mg, 2.5 mmol) were stirred under argon at 100 °C for 2.5 h. Then the mixture was crystallized from diethyl ether and recrystallized from diethyl ether:hexanes. Yield: 467 mg (74%). M.p.: 88-89 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 11.22 (1H, bs), 10.28 (1H, s), 7.10 (1H, d, *J* = 3.1 Hz), 7.01 (1H, s), 3.81-3.75 (8H, m), 3.70 (2H, s), 2.60 (3H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 191.6, 155.6, 152.4, 124.4, 122.3, 109.9, 66.7, 59.6, 55.8, 53.0. EI-HRMS calcd for $C_{13}H_{17}NO_4$ ($M^{\cdot+}$): 251.1158; found: 251.1159. Elemental analysis calcd (%) for $C_{13}H_{17}NO_4$: C 62.14, H 6.82, N 5.57; found: C 62.16, H 6.80, N 5.46.

General method for alkylation of salicylic aldehydes and hydroxyacetophenons (2a-2d, 2g, 2i-2k)

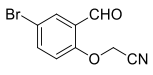
Salicylic aldehyde or hydroxyacetophenone (10 mmol), freshly grounded K_2CO_3 (2.8 g, 20 mmol), KI (0.83 g, 5.0 mmol) and dry DMF (50 ml) were places in a three neck flask and purged with argon. Then the mixture was cooled down to 10 °C and chloroacetonitrile (0.76 ml, 12 mmol) was added dropwise, over 5 minutes. The reaction mixture was stirred at given temperature for given time. Then it was slowly poured into ice-water (50 ml) and stirred for 2 hours. The precipitate were cooled to room temperature, filtered, washed with water and dried under reduce pressure to give expected product.

2-(2-Bromo-6-formylphenoxy)acetonitrile (2a)



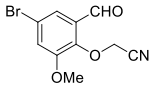
Prepared from 3-bromo-2-hydroxybenzaldehyde (**1a**, 2.01 g, 10 mmol). Reaction conditions: room temperature, 3 h. Yield: 2.16 g (90%). M.p.: 106-107 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 10.34 (1H, s), 7.88 (1H, q, *J* = 1.8 Hz), 7.86 (1H, q, *J* = 1.6 Hz), 7.28 (1H, t, *J* = 7.8 Hz), 4.96 (2H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 188.0, 155.4, 139.6, 131.6, 129.5, 127.5, 117.7, 114.2, 58.8. EI-HRMS calcd for $C_9H_6BrNO_2$ ($M^{\cdot+}$): 238.9582; found: 238.9588. Elemental analysis calcd (%) for $C_9H_6BrNO_2$: C 45.03, H 2.52, N 5.83, Br 33.29; found: C 44.88, H 2.54, N 5.84, Br 33.29.

2-(4-Bromo-2-formylphenoxy)acetonitrile (2b)



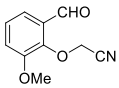
Prepared from 5-bromo-2-hydroxybenzaldehyde (**1b**, 2.01 g, 10 mmol). Reaction conditions: room temperature, 3h. Yield: 2.23 g (93%). M.p.: 109-110 °C (lit. 103-105 °C)[1]; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 10.36 (1H, s), 8.00 (1H, d, *J* = 2.6 Hz), 7.73 (1H, dd, J_1 = 8.9 Hz, J_2 = 2.6 Hz), 6.66 (1H, d, *J* = 8.7 Hz), 4.92 (2H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 187.0, 157.1, 138.4, 132.2, 127.0, 116.4, 114.6, 114.0, 54.0. EI-HRMS calcd for $C_9H_6BrNO_2$ ($M^{\cdot+}$): 238.9582; found: 238.9582.

2-(4-Bromo-2-formyl-6-methoxyphenoxy)acetonitrile (2c)



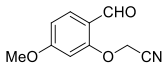
Prepared from 5-bromo-2-hydroxy-3-methoxybenzaldehyde (**1c**, 2.31 g, 10 mmol). Reaction conditions: room temperature, 3h. Yield: 2.32 g (86%). M.p.: 87-88 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 10.34 (1H, s), 7.60 (1H, d, *J* = 2.3 Hz), 7.30 (1H, d, *J* = 2.2 Hz), 4.96 (2H, s), 3.95 (3H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 187.3, 152.9, 147.1, 130.9, 122.7, 121.1, 119.0, 114.7, 57.9, 56.6. EI-HRMS calcd for $C_{10}H_8BrNO_3$ ($M^{\cdot+}$): 268.9688; found: 268.9684. Elemental analysis calcd (%) for $C_{10}H_8BrNO_3$: C 44.47, H 2.99, N 5.19, Br 29.59; found: C 44.30, H 2.81, N 5.20. Br 29.50.

2-(2-Formyl-6-methoxyphenoxy)acetonitrile (2d)



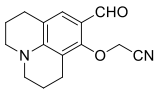
Prepared from 3-methoxy-2-hydroxybenzaldehyde (**1d**, 1.52 g, 10 mmol). Reaction conditions: room temperature, overnight. Yield: 1.58 g (85%). M.p.: 111-112 °C (lit. 117-119 °C)[1]; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 10.43 (1H, s), 7.48 (1H, dd, J_1 = 7.7 Hz, J_2 = 1.7 Hz), 7.28-7.19 (2H, m), 4.99 (2H, s), 3.95 (3H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 188.9, 152.1, 147.9, 130.1, 125.9, 120.0, 118.1, 115.0, 57.9, 56.2. EI-HRMS calcd for $C_{10}H_9NO_3$ ($M^{\cdot+}$): 191.0582; found: 191.0580.

2-(2-Formyl-5-methoxyphenoxy)acetonitrile (2g)



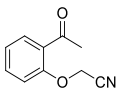
Prepared from 4-methoxy-2-hydroxybenzaldehyde (**1g**, 1.52 g, 10 mmol). Reaction conditions: room temperature, 3h. Purified by flash column chromatography (hexanes:ethyl acetate 1:1). Yield: 1.49 g (78%). M.p.: 100-101 °C (lit. 103-105 °C)[1]; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 10.25 (1H, s), 7.88 (1H, d, *J* = 8.7 Hz), 6.71 (1H, dd, J_1 = 8.7 Hz, J_2 = 2.0 Hz), 6.54 (1H, d, *J* = 2.0 Hz), 4.89 (2H, s), 3.91 (3H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 187.2, 166.0, 159.9, 131.7, 119.6, 114.3, 107.9, 99.6, 55.9, 53.8. EI-HRMS calcd for $C_{10}H_9NO_3$ ($M^{\cdot+}$): 191.0582; found: 191.0581.

2-((9-Formyl-2,3,6,7-tetrahydro-1H,5H-pyrido[3,2,1-ij]quinolin-8-yl)oxy)acetonitrile (2i)



Prepared from 8-hydroxy-2,3,6,7-tetrahydro-1H,5H-pirydo[3,2,1-ij]quinoline-9-carbaldehyde (**1i**, 2.31 g, 10 mmol). Reaction conditions: 50 °C, overnight. Yield: 1.25 g (49%). M.p.: 117-118 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 9.68 (1H, s), 7.19 (1H, s), 4.80 (2H, s), 3.30 (4H, q, *J* = 5.5 Hz), 2.85 (2H, t, *J* = 6.3 Hz), 2.73 (2H, t, *J* = 6.3 Hz), 1.99-1.92 (4H, m). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 187.4, 154.9, 148.9, 132.6, 117.6, 116.0, 115.7, 113.7, 58.6, 50.1, 49.7, 27.2, 21.2, 21.1, 20.6. EI-HRMS calcd for $C_{15}H_{16}N_2O_2$ ($M^{\cdot+}$): 256.1212; found: 256.1204. Elemental analysis calcd (%) for $C_{15}H_{16}N_2O_2$: C 70.29, H 6.29, N 10.93; found: C 70.29, H 6.09, N 11.4.

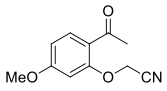
2-(2-Acetylphenoxy)acetonitrile (2j)



Prepared from 1-(2-hydroxyphenyl)ethanone (**1j**, 1.36 g, 10 mmol). Reaction conditions: 60 °C, 3 h. Purified by flash column chromatography (DCM). Yield: 1.57 g (89%). M.p.: 83-84 °C (lit. 82-83 °C)[2]; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.75 (1H, dd, J_1 = 7.7 Hz, J_2 = 1.7 Hz), 7.52 (1H,

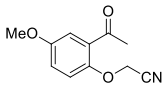
ddd, J_1 = 8.3 Hz, J_2 = 7.5 Hz, J_3 = 1.8 Hz), 7.16–7.04 (1H, m), 7.03 (1H, d, J = 8.3 Hz), 4.88 (2H, s), 2.61 (3H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 198.7, 155.2, 133.6, 130.9, 129.5, 123.3, 114.6, 113.2, 54.1, 31.6. EI-HRMS calcd for $C_{10}H_9NO_2$ ($M^{\cdot+}$): 175.0633; found: 175.0629.

2-(2-Acetyl-5-methoxyphenoxy)acetonitrile (2k)



Prepared from 1-(2-hydroxy-4-methoxyphenyl)ethanone (**1k**, 1.66 g, 10 mmol). Reaction conditions: room temperature, 3 h. Purified by flash column chromatography (hexanes:DCM 2:1). Yield: 1.29 g (63%). M.p.: 116-117 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.86 (1H, d, J = 8.8 Hz), 6.67 (1H, dd, J_1 = 8.8 Hz, J_2 = 2.2 Hz), 6.52 (1H, d, J = 2.1 Hz), 4.87 (2H, s), 3.88 (3H, s), 2.28 (3H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 196.6, 164.3, 157.4, 133.3, 122.0, 114.5, 107.6, 100.4, 55.8, 54.1, 31.5. ESI-HRMS calcd for $C_{11}H_{11}NO_3Na$ ($M+Na^+$): 228.0637; found: 228.0630. Elemental analysis calcd (%) for $C_{11}H_{11}NO_3$: C 64.38, H 5.40, N 6.83; found: C 64.43, H 5.29, N 6.79.

2-(2-Acetyl-4-methoxyphenoxy)acetonitrile (2l)



Prepared from 1-(2-hydroxy-5-methoxyphenyl)ethanone (**1l**, 1.66 g, 10 mmol). Reaction conditions: room temperature, overnight. Purified by flash column chromatography (hexanes:DCM 2:1). Yield: 1.54 g (75%). M.p.: 75-76 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.27 (1H, d, J = 3.0 Hz), 7.08-7.00 (2H, m), 4.82 (2H, s), 3.82 (3H, s), 2.61 (3H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 198.3, 155.5, 149.5, 130.5, 119.5, 116.1, 114.9, 114.8, 55.8, 55.4, 31.3. EI-HRMS calcd for $C_{11}H_{11}NO_3$ ($M^{\cdot+}$): 205.0739; found: 205.0736. Elemental analysis calcd (%) for $C_{11}H_{11}NO_3$: C 64.38, H 5.40, N 6.83; found: C 64.10, H 5.37, N 6.69.

Cyclization (Method A)

Alkylated salicylic aldehyde or hydroxyacetophenone (9.0 mmol), TBAHS (309 mg, 0.9 mmol), DCM (10 ml) and 50% aqueous solution of NaOH (10 ml) were placed in a flask and vigorously stirred at room temperature for 1 h. After reaction completion, the resulting mixture was cautiously poured into water (20 ml) and extracted with DCM. Combined organic phases were dried over $MgSO_4$ and concentrated. Crude product was purified by flash chromatography (DCM:hexanes,1:1).

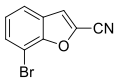
Cyclization (Method B)

Alkylated salicylic aldehyde or hydroxyacetophenone (9.0 mmol), freshly grounded K_2CO_3 (2.8 g, 20 mmol), TBAHS (153 mg, 0.45 mmol) and dry DMF (90 ml) were places in a three neck flask and purged with argon. The reaction mixture was stirred at 100 °C for 1 h, then it was cooled and extracted with DCM. Combined organic layers were dried over $MgSO_4$ and concentrated. The product was purified by the column chromatography in given eluting system.

One pot synthesis of cyanobeznofurans 3e, 3f, 3h (Method C)

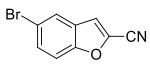
Salicylic aldehyde or hydroxyacetophenone (10 mmol) and freshly grounded K_2CO_3 (4.2 g, 30 mmol) were placed in a three neck flask and purged with argon. Then dry DMF (60 ml) and chloroacetonitrile (0.76 ml, 12 mmol) were added and the reaction mixture was stirred at 100 °C overnight. Then it was cooled and diluted with water and methylene chloride. The aqueous layer was extracted with methylene chloride and combined organic layers were dried over Na_2SO_4. Solvents were evaporated and the crude product was purified by the column chromatography in given eluting system.

7-Bromobenzofuran-2-carbonitrile (3a)



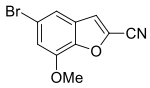
Prepared from **2a** (2.16 g, 9.0 mmol) according to method B. Yield: 1.04 g (52%). M.p.: 123-125 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.68 (1H, d, J = 7.7 Hz), 7.63 (1H, dd, J_1 = 7.9 Hz, J_2 = 0.4 Hz), 7.52 (1H, s), 7.26 (1H, t, J = 7.9 Hz). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 152.9, 131.4, 128.1, 126.7, 125.8, 121.7, 118.9, 111.1, 104.8. EI-HRMS calcd for C_9H_4BrNO ($M^{\cdot+}$): 220.9476; found: 220.9478. Elemental analysis calcd (%) for C_9H_4BrNO: C 48.68, H 1.82, N 6.31, Br 35.99; found: C 48.64, H 1.86, N 6.27, Br 36.01.

5-Bromobenzofuran-2-carbonitrile (3b)



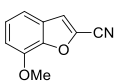
Prepared from (**2b**, 1.81 g, 9.0 mmol) according to method A. Yield: 659 mg (33%). Alternatively the title compound can be prepared according to method B. Purified by column chromatography (hexanes:DCM 2:3). Yield: 519 mg (26%). M.p.: 151-153 °C (lit. 147-149 °C)[3]; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.83 (1H, d, J = 2.0 Hz), 7.61 (1H, dd, J_1 = 8.9 Hz, J_2 = 2.0 Hz), 7.45 (1H, d, J = 8.9 Hz), 7.41 (1H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 154.3, 131.5, 128.5, 127.4, 125.1, 117.8, 117.5, 113.6, 111.2. EI-HRMS calcd for C_9H_4BrNO ($M^{\cdot+}$): 220.9476; found: 220.9477.

5-Bromo-7-methoxybenzofuran-2-carbonitrile (3c)



Prepared from **2c** (2.43 g, 9.0 mmol) according to method A. Yield: 544 mg (24%). Alternatively the title compound can be prepared according to method B. Purified by column chromatography (hexanes:DCM 2:3). Yield 45% (1.02 g). M.p.: 122-123 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.40 (1H, d, J = 2.5 Hz), 7.37 (1H, s), 7.07 (1H, d, J = 1.3 Hz), 4.02 (3H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 146.0, 144.4, 128.4, 128.3, 118.0, 117.7, 116.8, 113.5, 111.0, 56.6. EI-HRMS calcd for $C_{10}H_6BrNO_2$ ($M^{\cdot+}$): 250.9582; found: 250.9579. Elemental analysis calcd (%) for $C_{10}H_6BrNO_2$: C 47.65, H 2.40, N 5.56, Br 31.70; found: C 47.87, H 2.28, N 5.55, Br 31.72.

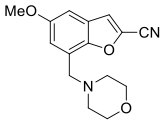
7-Methoxybenzofuran-2-carbonitrile (3d)



Prepared from **2d** (1.72 g, 9.0 mmol) according to method A. Yield: 747 mg (48%). Alternatively, the title compound can be prepared according to method B. Purified by column chromatography (DCM). Yield: 1.12 g (72%). M.p.: 103-105 °C (lit. 102-104 °C)[3]; ^{1}H NMR

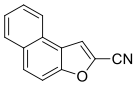
($CDCl_3$, 500 MHz): δ 7.44 (1H, s), 7.30–7.25 (2H, m), 6.98 (1H, dd, J_1 = 7.8 Hz, J_2 = 1.1 Hz), 4.03 (3H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 145.7, 145.5, 127.5, 127.2, 125.4, 118.7, 114.3, 111.6, 109.7, 56.2. EI-HRMS calcd for $C_{10}H_7NO_2$ ($M^{\cdot+}$): 173.0477; found: 173.0474.

5-Methoxy-7-(morpholinomethyl)benzofuran-2-carbonitrile (3e)



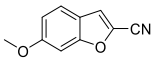
Prepared from **1e** (2.51 g, 10 mmol) according to method C. Purified by column chromatography (DCM:acetone 9:1 → 4:1). Yield: 2.72 g (51%). M.p.: 98-100 °C; ^1H NMR (500 MHz, $CDCl_3$): δ 7.38 (1H, s), 7.17 (1H, s), 6.96 (1H, d, J = 2.5 Hz), 3.85 (3H, s), 3.79 (2H, s), 3.75-3.71 (4H, m), 2.54 (4H, s). ^{13}C NMR (126 MHz, $CDCl_3$): δ 157.2, 149.9, 127.6, 126.0, 118.8, 118.5, 111.9, 102.1, 66.9, 56.2, 55.9, 53.5. ESI-HRMS calcd for $C_{15}H_{17}N_2O_3$ ($M+H^+$): 273.1239; found: 273.1245.

Naphtho[2,1-b]furan-2-carbonitrile (3f)



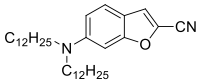
Prepared from 1-formyl-2-hydroxynaphtalene (**1f**, 1.72 g, 10 mmol) according to method C. Purified by column chromatography (DCM). Yield: 617 mg (32%). M.p.: 89-90 °C (lit. 88-90 °C)[3]; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.11 (1H, bd, J = 8.1 Hz), 7.97 (1H, bd, J = 8.4 Hz), 7.92 (1H, bd, J = 9.1 Hz), 7.90 (1H, d, J = 1.0 Hz), 7.69–7.65 (1H, m), 7.64 (1H, dd, J_1 = 9.1 Hz, J_2 = 0.7 Hz), 7.60–7.56 (1H, m). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 154.2, 130.7, 130.0, 129.1, 127.8, 127.3, 126.5, 125.9, 123.2, 121.5, 117.3, 112.2, 112.1. EI-HRMS calcd for $C_{13}H_7NO$ ($M^{\cdot+}$): 193.0528; found: 193.0526.

6-Methoxybenzofuran-2-carbonitrile (3g)



Prepared from **2g** (1.72 g, 9.0 mmol) according to method B. Reaction time: 3h. Purified by column chromatography (hexsanes:DCM 3:2). Yield: 982 mg (63%). M.p.: 80-81 °C (lit. 78-80 °C)[3]; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.52 (1H, d, J = 8.5 Hz), 7.38 (1H, d, J = 0.5 Hz), 7.01-6.98 (2H, m), 3.88 (3H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 161.2, 157.2, 126.3, 122.7, 118.6, 118.5, 114.9, 112.2, 95.4, 55.8. EI-HRMS calcd for $C_{10}H_7NO_2$ ($M^{\cdot+}$): 173.0477; found: 173.0478.

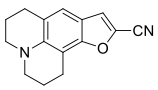
6-(Didodecylamino)benzofuran-2-carbonitrile (3h)



Prepared from 4-didocylamino-2-hydroxybenzaldehyde (**1h**, 4.73 g, 10 mmol) according to method C. Purified by column chromatography (hexanes:ethyl acetate 95:5). Yield: 3.07 g (62%). M.p.: 34-35 °C; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.38 (1H, d, J = 8.9 Hz), 7.27 (1H, s), 6.72 (1H, d, J = 8.8 Hz), 6.62 (1H, s), 3.34-3.28 (4H, m), 1.65-1.56 (4H, m), 1.36-1.24 (36H, m), 0.91-0.86 (6H, m). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 158.7, 149.6, 122.4, 118.9, 113.1, 111.4, 92.8, 51.6, 31.9, 29.6, 29.6, 29.6, 29.6, 29.5, 29.3, 27.1, 22.7, 14.1. HRMS calcd for $C_{33}H_{54}N_2O$ ($M^{\cdot+}$):

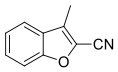
494.4236; found: 494.4226. Elemental analysis calcd (%) for $C_{33}H_{54}N_2O$: C 80.10, H 11.00, N 5.66; found: C 80.13, H 10.96, N 5.65.

2,3,6,7-tetrahydro-1H,5H-furo[2,3-f]pyrido[3,2,1-ij]quinoline-10-carbonitrile (3i)



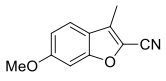
Prepared from **2i** (2.30 g, 9.0 mmol) according to method A. Reaction time: 2 h. Yield: 1.26 g (59%). M.p.: 120-121 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.20 (1H, s), 6.99 (1H, s), 3.23 (4H, dd, J_1 = 11.8 Hz, J_2 = 6.6 Hz), 2.90 (2H, t, J = 6.5 Hz), 2.83 (2H, t, J = 6.4 Hz), 2.06-1.94 (4H, m). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 154.6, 143.6, 123.9, 121.1, 118.9, 118.6, 113.9, 113.4, 103.3, 50.2, 49.8, 28.3, 21.9, 20.8, 20.5. HRMS calcd for $C_{15}H_{15}N_2O$ ($M+H^+$): 239.1184; found: 239.1184. Elemental analysis calcd (%) for $C_{15}H_{14}N_2O$: C 75.61, H 5.92, N 11.76; found: C 75.35, H 6.09, N 11.48.

3-methylbenzofuran-2-carbonitrile (3j)



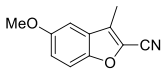
Prepared from **2j** (1.58 g, 9.0 mmol) according to method A. Reaction time: 2 h. Yield: 1.37 g (97%). M.p.: 66-67 °C (lit. 71-72 °C)[4]; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.60 (1H, bd, J = 7.9 Hz), 7.50 (2H, bd, J = 3.9 Hz), 7.38–7.33 (1H, m), 2.46 (3H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 155.4, 129.7, 128.4, 126.9, 125.0, 123.9, 120.9, 112.1, 111.9, 8.9. EI-HRMS calcd for $C_{10}H_7NO$ ($M^{\cdot+}$): 157.0528; found: 157.0522.

6-methoxy-3-methylbenzofuran-2-carbonitrile (3k)



Prepared from **2k** (1.85 g, 9.0 mmol) according to method A. Yield: 960 mg (57%). M.p.: 86-87 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.44 (1H, dd, J_1 = 8.2 Hz, J_2 = 0.9 Hz), 6.99-6.95 (2H, m), 3.87 (3H, s), 2.41 (3H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 161.2, 156.8, 130.0, 124.1, 121.1, 120.2, 114.1, 112.2, 95.5, 55.8, 8.8. EI-HRMS calcd for $C_{11}H_9NO_2$ ($M^{\cdot+}$): 187.0633; found:187.0630.

5-methoxy-3-methylbenzofuran-2-carbonitrile (3l)



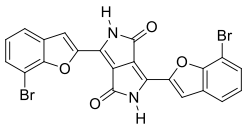
Prepared from **2l** (1.85 g, 9.0 mmol) according to method A. Yield: 1.50 g (89%). M.p.: 110-111 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.38 (1H, d, J = 9.1 Hz), 7.10 (1H, dd, J_1 = 9.1 Hz, J_2 = 2.7 Hz), 6.96 (1H, d, J = 2.7 Hz), 3.87 (3H, s), 2.42 (3H, s). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 156.8, 150.4, 129.6, 127.4, 125.5, 118.1, 112.7, 111.9, 101.9, 55.9, 8.8. EI-HRMS calcd for $C_{11}H_9NO_2$ ($M^{\cdot+}$): 187.0633; found: 187.0637. Elemental analysis calcd (%) for $C_{11}H_9NO_2$: C 70.58, H 4.85, N 7.48; found: C 70.43, H 5.04, N 7.38.

General procedure for synthesis of DPP (4a-h and 4j-k)

Tert-amyl alcohol (8 ml), sodium (250 mg, 11 mmol) and a catalytic amount of $FeCl_3$ were placed in a three necked flask. The mixture was refluxed under an argon atmosphere until sodium has completely reacted. Then the reaction mixture was cooled to 90 °C and cyanobenzofuran (5.0 mmol) was added.

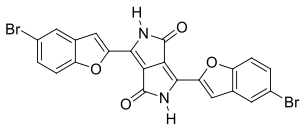
The mixture was then heated to 110 °C and diisopropyl succinate (0.37 ml, 2.2 mmol) was added dropwise. After 16h of reaction at 110 °C, the mixture was quenched by the addition 20 ml of mixture water/methanol/acetic acid (1:1:1). The resulting suspension was refluxed for 1 h. Unless otherwise stated, the precipitate of the obtained pigment was filtered while still hot, washed several times with hot water and methanol and dried under vacuum.

3,6-bis(7-bromobenzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (4a)



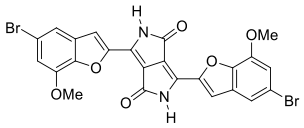
Prepared from **3a** (1.11 g, 5.0 mmol). Yield: 601 mg (52%). M.p.: decomp. > 390 °C. The NMR spectra of the product could not be obtained due to the low solubility. EI-HRMS calcd for $C_{22}H_{10}Br_2N_2O_4$ ($M^{\cdot+}$): 523.9007; found: 523.8995.

3,6-bis(5-bromobenzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (4b)



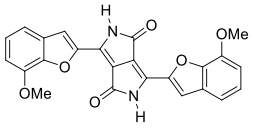
Prepared from **3b** (1.11 g, 5.0 mmol). Yield: 381 mg (33%). M.p.: > 400 °C. The NMR spectra of the product could not be obtained due to the low solubility. EI-HRMS calcd for $C_{22}H_{10}Br_2N_2O_4$ ($M^{\cdot+}$): 523.9007; found: 523.8996. Elemental analysis calcd (%) for $C_{22}H_{10}Br_2N_2O_4$: C 50.22, H 1.92, N 5.32, Br 30.37; found: C 50.03, H 1.94, N 5.15, Br 30.20.

3,6-bis(5-bromo-7-methoxybenzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (4c)



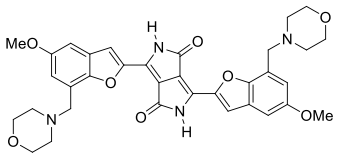
Prepared from **3c** (1.26 g, 5.0 mmol). Yield: 734 mg (57%). M.p.: decomp. > 340 °C. The NMR spectra of the product could not be obtained due to the low solubility. EI-HRMS calcd for $C_{24}H_{14}Br_2N_2O_6$ ($M^{\cdot+}$): 583.9219; found: 583.9216. Elemental analysis calcd (%) for $C_{24}H_{14}Br_2N_2O_6$: C 49.17, H 2.41, N 4.78, Br 27.26; found: C 48.93, H 2.43, N 4.89, Br 27.17.

3,6-bis(7-methoxybenzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (4d)



Prepared from **3d** (865 mg, 5.0 mmol). Yield: 339 mg (36%). M.p.: > 400 °C. The NMR spectra of the product could not be obtained due to the low solubility. EI-HRMS calcd for $C_{24}H_{16}N_2O_6$ ($M^{\cdot+}$): 428.0998; found: 428.1008.

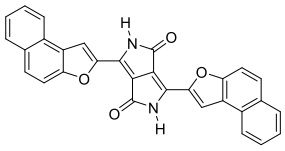
3,6-bis(5-methoxy-7-(morpholinomethyl)benzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (4e)



Prepared from **3e** (1.36 g, 5.0 mmol). Yield: 579 mg (42%). M.p: > 400°C. The NMR spectra of the product could not be obtained due

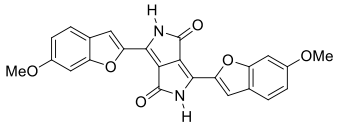
to the low solubility. EI-HRMS calcd for $C_{34}H_{34}N_4O_8$ ($M^{\cdot+}$): 626.2377; found: 626.2383.

3,6-bis(naphtho[2,1-b]furan-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (4f)



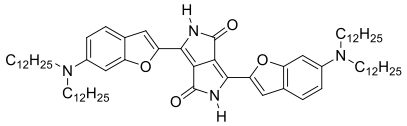
Prepared from **3f** (965 mg, 5.0 mmol). Yield: 742 mg (72%). M.p: > 400°C. The NMR spectra of the product could not be obtained due to the low solubility. EI-HRMS calcd for $C_{30}H_{16}N_2O_4$ ($M^{\cdot+}$): 468.1110; found: 468.1113.

3,6-bis(6-methoxybenzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (4g)



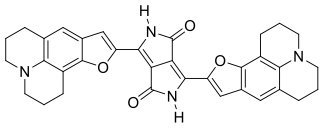
Prepared from **3g** (865 mg, 5.0 mmol). Yield: 325 mg (28%). M.p.: > 400 °C. The NMR spectra of the product could not be obtained due to the low solubility. HRMS calcd for $C_{24}H_{16}N_2O_6$ ($M^{\cdot+}$): 428.1008; found: 428.0999. Elemental analysis calcd (%) for $C_{24}H_{16}N_2O_6$: C 67.29, H 3.76, N 6.54; found: C 67.16, H 4.00, N 6.45.

3,6-bis(6-(didodecylamino)benzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (4h)



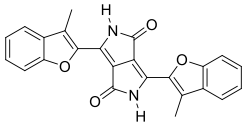
Prepared from **3h** (2.48 g, 5.0 mmol). Yield: 353 mg (15%). After quenching the reaction mixture was concentrated almost to dryness, 20 ml of cyclohexane was added and the resulting solution was filtered through Celite pad. More cyclohexane was added until all coloured compounds were washed out, except of green compound on the top of Celite pad. Then, collecting flask was replaced and Celite pad was washed with DCM. Green compound was collected, evaporated and crystallized from pyridine. M.p.: 202-203 °C; ^{1}H NMR (C_6D_6, 500 MHz, 70 °C): δ 8.45 (2H, s), 7.35 (2H, s), 7.23 (2H, d, *J* = 9.0 Hz), 6.74 (2H, s), 6.65 (2H, d, *J* = 9.0 Hz), 3.19 (8H, t, *J* = 7.5 Hz), 1.63-1.54 (8H, m), 1.37-1.25 (56H, m), 0.97-0.87 (16H, m), 0.46-0.39 (12H, m). ^{13}C NMR ($CDCl_3$, 126 MHz, 70 °C): δ signals from aromatic carbon atoms were not detected, 51.9, 32.3, 30.1, 30.1, 30.0, 30.0, 29.9, 29.7, 27.7, 27.5, 23.0, 14.2. ESI-HRMS calcd for $C_{70}H_{111}N_4O_4$ ($M+H^+$): 1071.8605; found: 1071.8608. Elemental analysis calcd (%) for $C_{70}H_{110}N_4O_4$: C 78.45, H 10.35, N 5.23; found: C 78.44, H 10.21, N 5.04.

3,6-bis(2,3,6,7-tetrahydro-1H,5H-furo[2,3-f]pyrido[3,2,1-ij]quinolin-10-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (4i)



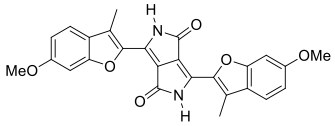
Prepared from **3i** (1.19 g, 5.0 mmol). Yield: 847 mg (69%). M.p.: decomp. > 250 °C. The NMR spectra of the product could not be obtained due to the low solubility. EI-HRMS calcd for $C_{34}H_{30}N_4O_4$ ($M^{\cdot+}$): 558.2267; found: 558.2282.

3,6-bis(3-methylbenzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (4j)



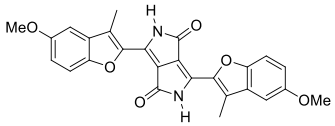
Prepared from **3j** (789 mg, 5.0 mmol). Yield: 497 mg (57%). M.p.: decomp. > 315 °C. The NMR spectra of the product could not be obtained due to the low solubility. EI-HRMS calcd for $C_{24}H_{16}N_2O_4$ ($M^{\cdot+}$): 396.1110; found: 396.1117. Elemental analysis calcd (%) for $C_{24}H_{16}N_2O_4$: C 72.72, H 4.07, N 7.07; found: C 72.67, H 4.35, N 6.78.

3,6-bis(6-methoxy-3-methylbenzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (4k)



Prepared from **3k** (936 mg, 5.0 mmol). Yield: 532 mg (53%). M.p.: decomp. > 205 °C. The NMR spectra of the product could not be obtained due to the low solubility. ESI-HRMS calcd for $C_{26}H_{20}N_2O_6Na$ ($M+Na^+$): 479.1219; found: 479.1208.

3,6-bis(5-methoxy-3-methylbenzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione(4l)

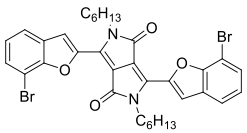


Prepared from **3k** (936 mg, 5.0 mmol). Yield: 215 mg (21%). M.p.: decomp. > 320 °C. The NMR spectra of the product could not be obtained due to the low solubility. HRMS calcd for $C_{26}H_{20}N_2O_6$ ($M^{\cdot+}$): 456.1321; found: 456.1312.

General method of alkylation of DPP (5a-h and 5j-k)

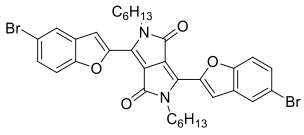
A mixture of DPP (1 mmol), TBAHS (17 mg, 0.05 mmol), K_2CO_3 (2.07 g, 15 mmol) in DMF (25 ml) was heated to 120 °C under argon atmosphere. Then *n*-bromohexane (1.4 ml, 10 mmol) or 2-(2-(2-(methoxy)ethoxy)ethoxy)ethyl chloride (19.8 ml, 10 mmol) was added dropwise. The reaction mixture was stirred overnight at 120 °C, then cooled down. Unless otherwise specified, the mixture was extracted with DCM and combined organic layers were dried over $MgSO_4$. Solvents were evaporated and the final product was purified by the column chromatography in given eluting system.

3,6-bis(7-bromobenzofuran-2-yl)-2,5-dihexyl-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (5a)



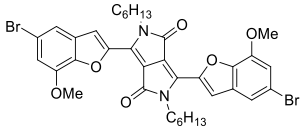
Prepared from **4a** (526 mg, 1.0 mmol) and *n*-bromohexane. Purified by column chromatography (DCM). Yield: 417 mg (60%). M.p.: 286-288 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.78 (2H, s), 7.62 (2H, d, *J* = 7.7 Hz), 7.52 (2H, d, *J* = 7.5 Hz), 7.16 (2H, t, *J* = 7.8 Hz), 4.31-4.25 (4H, m), 1.86-1.78 (4H, m), 1.57-1.49 (4H, m), 1.41-1.29 (8H, m), 0.89 (6H, t, *J* = 7.0 Hz). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 160.7, 152.8, 145.8, 133.9, 130.1, 129.2, 125.3, 121.7, 116.5, 109.4, 104.1, 43.3, 31.7, 30.7, 26.7, 22.6, 14.0. EI-HRMS calcd for $C_{34}H_{34}Br_2N_2O_4$ ($M^{\cdot+}$): 692.0885; found: 692.0876. Elemental analysis calcd (%) for $C_{34}H_{34}$ $Br_2N_2O_4$: C 58.80, H 4.93, Br 23.01, N 4.04; found: C 58.61, H 4.79, Br 23.03, N 3.92.

3,6-bis(5-bromobenzofuran-2-yl)-2,5-dihexyl-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (5b)



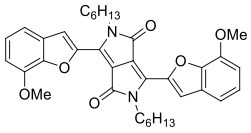
Prepared from **4b** (526 mg, 1.0 mmol) and *n*-bromohexane. Crystallization from DCM. Yield: 694 mg (70%). M.p.: 276-277 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.69 (2H, s), 7.84 (2H, d, J = 1.6 Hz), 7.53 (2H, dd, J_1 = 8.7 Hz, J_2 = 1.8 Hz), 7.40 (2H, d, J = 8.7 Hz), 4.24 (4H, t, J = 7.6 Hz), 1.77 (4H, p, J = 7.7 Hz), 1.50-1.42 (4H, m), 1.41-1.34 (8H, m), 0.90 (6H, t, J = 7.0 Hz). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 160.7, 154.3, 146.4, 134.1, 130.4, 130.0, 125.1, 117.4, 115.4, 112.9, 109.5, 42.8, 31.4, 30.3, 26.6, 22.6, 14.0. EI-HRMS calcd for $C_{34}H_{34}Br_2N_2O_4Na$ ($M^{\cdot+}$): 715.0783; found: 715.0781.

3,6-bis(5-bromo-7-methoxybenzofuran-2-yl)-2,5-dihexyl-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (5c)



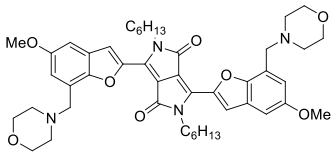
Prepared from **4c** (586 mg, 1.0 mmol) and *n*-bromohexane. Purified by column chromatography (DCM:hexanes 1:1 → 3:1). Yield: 679 mg (90%). M.p.: 245-246 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.62 (2H, s), 7.41 (2H, d, J = 1.4 Hz), 6.99 (2H, d, J = 1.3 Hz), 4.26-4.21 (4H, m), 4.00 (6H, s), 1.81-7.74 (4H, m), 1.52-1.44 (4H, m), 1.40-1.28 (8H, m), 0.98-0.87 (6H, m). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 160.7, 146.2, 145.9, 144.1, 134.1, 130.7, 117.2, 116.9, 115.3, 112.5, 109.2, 56.3, 43.0, 31.4, 30.4, 26.6, 22.6, 14.0. EI-HRMS calcd for $C_{36}H_{38}Br_2N_2O_6$ ($M^{\cdot+}$): 752.1097; found: 752.1094.

2,5-dihexyl-3,6-bis(7-methoxybenzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (5d)



Prepared from **4d** (428 mg, 1 mmol) and *n*-bromohexane. Purified by column chromatography (DCM:hexanes 1:1). Yield: 364 mg (61%). M.p.: 263-264 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.71 (2H, s), 7.29 (2H, d, J = 7.6 Hz), 7.21 (2H, t, J = 7.8 Hz), 6.89 (2H, d, J = 7.7 Hz), 4.30-4.24 (4H, m), 4.01 (6H, s), 1.84-1.76 (4H, m), 1.53-1.46 (4H, m), 1.41-1.29 (8H, m), 0.90 (6H, t, J = 7.0 Hz). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 160.9, 145.6, 145.5, 145.2, 134.3, 129.8, 124.6, 116.4, 114.6, 109.1, 108.8, 56.0, 43.0, 31.4, 30.4, 26.6, 22.7, 14.0. ESI-HRMS calcd for $C_{36}H_{40}N_2O_6Na$ ($M+Na^+$): 619.2784; found: 619.2783. Elemental analysis calcd (%) for $C_{36}H_{40}N_2O_6$: C 72.46, H 6.76, N 4.69; found: C 72.23, H 6.71, N 4.58.

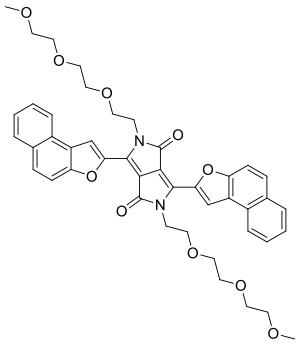
2,5-dihexyl-3,6-bis(5-methoxy-7-(morpholinomethyl)benzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (5e)



Prepared from **4e** (627 mg, 1 mmol) and *n*-bromohexane. Purified by column chromatography (DCM → DCM:MeOH 99:1) and crystallization form DCM:acetone. Yield: 628 mg (79%). M.p.: 231-232 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.71 (2H, s), 7.77 (2H, s), 7.02 (2H, d, J = 2.1 Hz), 4.28 (4H, t, J = 7.3 Hz), 3.87 (6H, s), 3.79 (4H, s), 3.75-3.71 (8H, m), 2.54 (8H, s), 1.82 (4H, p, J = 7.4 Hz), 1.52-1.44 (4H, m), 1.40-

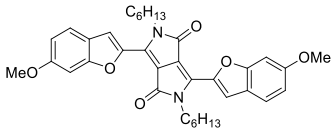
1.30 (8H, m), 0.89 (6H, t, *J* = 6.9 Hz). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 160.9, 156.9, 150.0, 145.9, 134.1, 128.6, 122.6, 117.9, 116.5, 108.8, 102.4, 67.0, 56.8, 55.9, 53.8, 42.9, 31.7, 30.6, 26.9, 22.7, 14.0. EI-HRMS calcd for $C_{46}H_{58}N_4O_8$ ($M^{\cdot+}$): 794.4255; found: 794.4226. Elemental analysis calcd (%) for $C_{46}H_{58}N_2O_8$: C 69.50, H 7.35, N 7.05; found: C 69.35, H 7.09, N 7.06.

2,5-bis(2-(2-(2-methoxyethoxy)ethoxy)ethyl)-3,6-bis(naphtho[2,1-b]furan-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (5f)



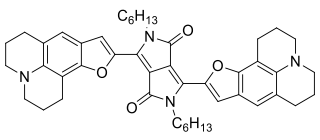
Prepared from **4f** (468 mg, 1.0 mmol) and 2-(2-(2-(methoxy)ethoxy)ethoxy)ethyl chloride. Purified by column chromatography (DCM:acetone 4:1) and crystallization from toluene. Yield: 350 mg (46%). M.p.: 223–224°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.13 (2H, s), 8.29 (2H, d, *J* = 8.1 Hz), 7.86 (2H, d, *J* = 8.1 Hz), 7.75 (2H, d, *J* = 9.0 Hz), 7.67-7.62 (2H, m), 7.58 (2H, d, *J* = 8.9 Hz), 7.59-7.52 (2H, m), 4.58 (4H, t, *J* = 6.2 Hz), 3.88 (4H, t, *J* = 6.2 Hz), 3.72-3.68 (4H, m), 3.58-3.53 (4H, m), 3.50-3.46 (4H, m), 3.38-3.33 (4H, m), 3.25 (6H, s). ^{13}C NMR (126 MHz, $CDCl_3$): δ 160.8, 153.8, 145.0, 133.6, 130.6, 129.0, 128.8, 127.5, 127.3, 125.6, 124.2, 124.1, 115.1, 111.8, 108.4, 71.8, 70.7, 70.6, 70.5, 69.8, 58.9, 42.0. ESI-HRMS calcd for: $C_{44}H_{44}N_2O_{10}Na$ ($M+Na^+$): 783.2894; found: 783.2874.

2,5-dihexyl-3,6-bis(6-methoxybenzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (5g)



Prepared from **4g** (428 mg, 1.0 mmol) and *n*-bromohexane. Combined organic layers were washed with brine and dried over $MgSO_4$. The mixture was concentrated to ¼ volume and the precipitate was filtered and washed with DCM. Yield: 448 mg (75%). M.p.: 279-280 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.69 (2H, s), 7.56 (2H, d, *J* = 8.4 Hz), 6.99-6.92 (4H, m), 4.27-4.20 (4H, m), 3.89 (6H, s), 1.82-1.74 (4H, m), 1.51-1.43 (4H, m), 1.42-1.29 (8H, m), 0.93-0.88 (6H, m). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 160.8, 160.5, 157.1, 145.0, 133.6, 122.9, 121.7, 116.4, 114.0, 107.9, 95.2, 55.8, 42.7, 31.5, 30.2, 26.6, 22.6, 14.1. EI-HRMS calcd for $C_{36}H_{40}N_2O_6$ ($M^{\cdot+}$): 596.2886; found: 596.2880. Elemental analysis calcd (%) for $C_{36}H_{40}N_2O_6$: C 72.46, H 6.76, N 4.69; found: C 72.24, H 6.97, N 4.52.

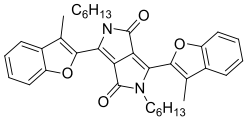
2,5-dihexyl-3,6-bis(2,3,6,7-tetrahydro-1H,5H-furo[2,3-f]pyrido[3,2,1-ij]quinolin-10-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (5i)



Prepared from **4i** (559 mg, 1.0 mmol) and *n*-bromohexane. Purified by column chromatography (hexanes:DCM 1:3) and crystallized from pyridine. Yield: 516 mg (71%). M.p.: 250-252 °C; ^{1}H NMR (C_6D_6, 500 MHz, 70 °C): δ 9.21 (2H, s), 6.83 (2H, s), 4.42-4.37 (4H, m), 2.84-2.76 (12H, m), 2.54 (4H, t, *J* = 6.5 Hz), 1.92 (4H, p, *J* = 7.5 Hz), 1.73 (4H, p, *J* = 5.5 Hz), 1.64 (4H, p, *J* = 5.5 Hz), 1.50 (4H, p, *J* = 7.5

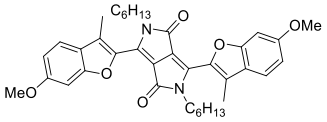
Hz), 1.36-1.25 (8H, m), 0.86 (6H, t, J = 7.0Hz). ^{13}C NMR (C_6D_6, 126 MHz, 70 °C): δ signals from aromatic carbon atoms were not detected, 50.5, 50.1, 43.0, 32.1, 31.1, 28.5, 27.2, 25.4, 22.9, 22.6, 21.5, 21.2, 14.1. EI-HRMS calcd for $C_{46}H_{54}N_4O_4$ ($M^{\cdot+}$): 726.4145; found: 726.4160. Elemental analysis calcd (%) for $C_{46}H_{54}N_4O_4$: C 76.00, H 7.49, N 7.71; found: C 76.16, H 7.60, N 7.90.

2,5-dihexyl-3,6-bis(3-methylbenzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (5j)



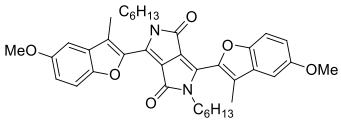
Prepared from **4j** (396 mg, 1.0 mmol) and *n*-bromohexane. Purified by column chromatography (DCM). Yield: 136 mg (24%). M.p.: 166-167 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.69 (2H, d, J = 7.8 Hz), 7.48 (2H, d, J = 8.2 Hz), 7.43 (2H, t, J = 8.2 Hz), 7.34 (2H, t, J = 7.4 Hz), 3.96-3.89 (4H, m), 2.55 (6H, s), 1.64 (4H, p, J = 7.3 Hz), 1.33-1.19 (12H, m), 0.79-0.84 (6H, m). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 160.8, 155.2, 140.0, 135.8, 129.7, 127.1, 125.6, 123.3, 120.9, 111.4, 110.2, 42.5, 31.3, 29.5, 26.4, 22.5, 13.9, 10.1. ESI-HRMS calcd for $C_{36}H_{40}N_2O_4Na$ ($M+Na^+$): 587.2886; found: 587.2879. Elemental analysis calcd (%) for $C_{36}H_{40}N_2O_4$: C 76.57, H 7.14, N 4.96; found: C 76.52, H 7.18, N 4.86.

2,5-dihexyl-3,6-bis(6-methoxy-3-methylbenzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (5k)



Prepared from **4k** (456 mg, 1.0 mmol) and *n*-bromohexane. Purified by column chromatography (DCM:hexanes 1:1). Yield: 225 mg (36%). M.p.: 164-165 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.56-752 (2H, m), 6.98-6.94 (4H, m), 3.95-3.88 (10H, m), 2.25 (6H, s), 1.64 (4H, p, J = 7.2 Hz), 1.33-1.21 (12H, m), 0.83 (6H, t, J = 6.8 Hz). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 160.9, 160.3, 156.5, 139.4, 135.3, 126.0, 123.3, 121.2, 113.0, 109.4, 95.3, 55.8, 42.5, 31.3, 29.4, 26.4, 22.5, 14.0, 10.23. EI-HRMS calcd for $C_{38}H_{44}N_2O_6$ ($M^{\cdot+}$): 624.3199; found: 624.3206. Elemental analysis calcd (%) for $C_{38}H_{44}N_2O_6$: C 73.05, H 7.10, N 4.48; found: C 73.03, H 7.26, N 4.50.

2,5-dihexyl-3,6-bis(5-methoxy-3-methylbenzofuran-2-yl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (5l)

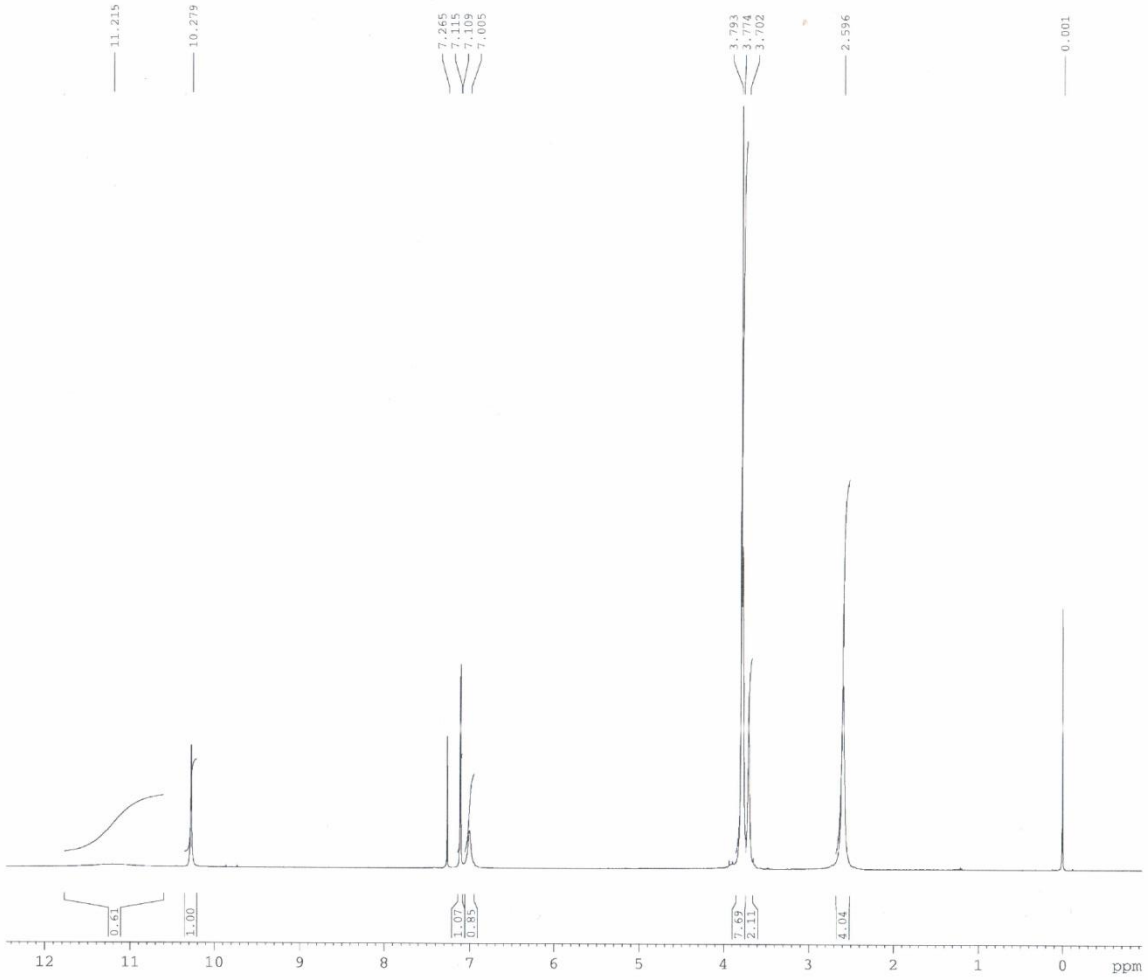


Prepared from **4l** (456 mg, 1.0 mmol) and *n*-bromohexane**.** Purified by column chromatography (DCM:hexanes 1:1) and crystallization from *i*-PrOH. Yield: 131 mg (21%). M.p.: 119-120 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.37 (2H, d, J = 8.8 Hz), 7.07 (2H, d, J = 2.6 Hz), 7.04 (2H, dd, J_1 = 8.9 Hz, J_2 = 2.6 Hz), 3.93-3.88 (10H, m), 2.51 (6H, s), 1.62 (4H, p, J = 7.2 Hz), 1.32-1.19 (12H, m), 1.82 (6H, t, J = 6.9 Hz). ^{13}C NMR ($CDCl_3$, 126 MHz): δ 160.8, 156.4, 150.3, 140.8, 135.7, 130.2, 125.5, 116.7, 112.0, 110.0, 102.3, 56.0, 42.5, 31.3, 29.4, 26.4, 22.5, 13.9, 10.2. HRMS calcd for $C_{38}H_{44}N_2O_6$ ($M^{\cdot+}$): 624.3199; found: 624.3209. Elemental analysis calcd (%) for $C_{38}H_{44}N_2O_6$: C 73.05, H 7.10, N 4.48; found: C 73.12, H 7.04, N 4.34.

Table S1. Crystallographic data for **5j**. CCDC No. 1515317

Chemical formula	$C_{36}H_{40}N_2O_4$	
Formula weight	564.70 g/mol	
Temperature	273(2) K	
Wavelength	1.54178 Å	
Crystal size	0.130 x 0.142 x 0.222 mm	
Crystal habit	orange plate	
Crystal system	monoclinic	
Space group	P1 21/c1	
Unit cell dimensions	a = 11.882(2) Å	α = 90°
	b = 6.1567(13) Å	β = 99.617(15)°
	c = 22.083(4) Å	γ = 90°
Volume	1592.8(6) $Å^3$	
Z	2	
Diffractometer	Bruker APEX-II CCD	
Radiation source	fine-focus sealed tube, CuK_α	
Reflections collected	12084	
Independent reflections	2696 [R(int) = 0.1045]	
Tmin, Tmax	0.902, 0.924	
Absorption correction	numerical	
Refinement method	Full-matrix least-squares on F^2	
Function minimized	$\Sigma\, w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	2696 / 0 / 200	
Goodness-of-fit on F^2	0.938	
Δ/σ_{max}	0.056	
Final R indices	1166 data; I>2σ(I)	R1 = 0.0736, wR2 = 0.1614
	all data	R1 = 0.1821, wR2 = 0.1933
Largest diff. peak and hole	0.310 and -0.173 $eÅ^{-3}$	

The crystals of **5j** suitable for X-ray crystallography were obtained by slow evaporation of solution of **5j** in dichloromethane/cyclohexane mixture. The X-ray intensity data were measured on a Bruker APEX-II CCD system equipped with a graphite monochromator and a CuK_α fine-focus sealed tube (λ = 1.54178 Å). A total of 2291 frames were collected. The total exposure time was 44.55 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. Data were corrected for absorption effects using the numerical method (SADABS). The structure was solved and refined using the Bruker SHELXTL Software Package.



BRUKER

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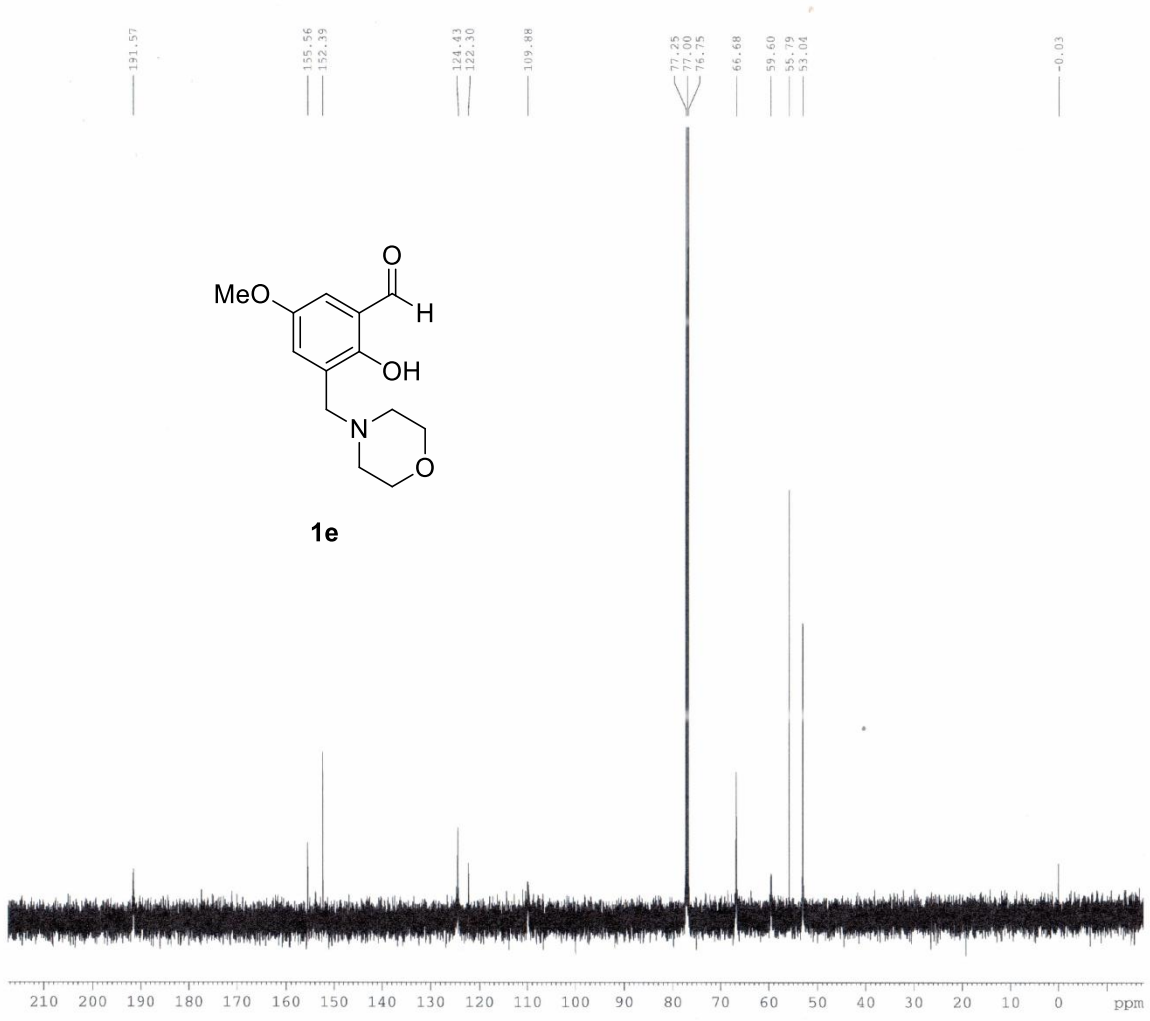

1e

BRUKER

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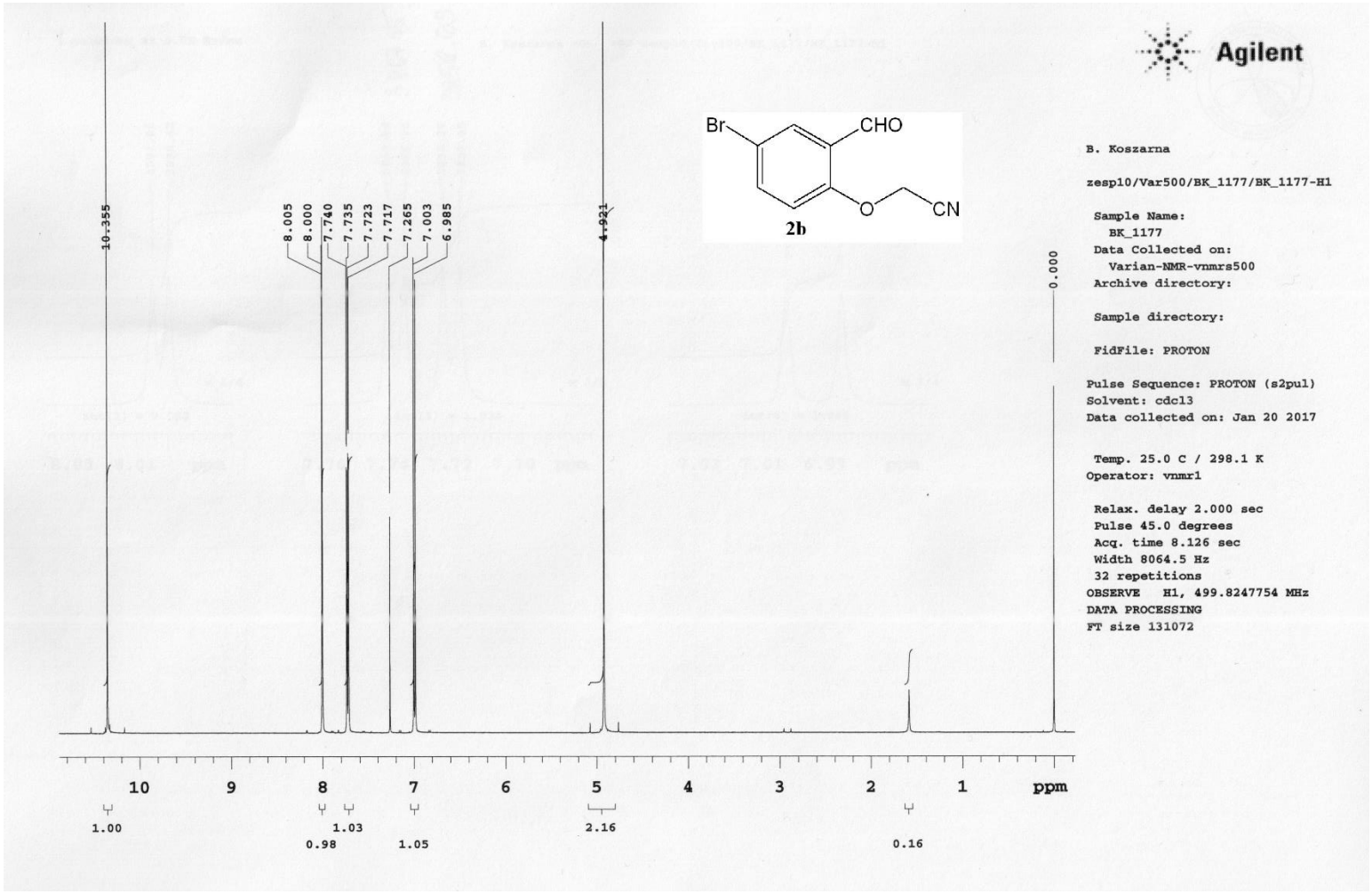
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B. Koszarna
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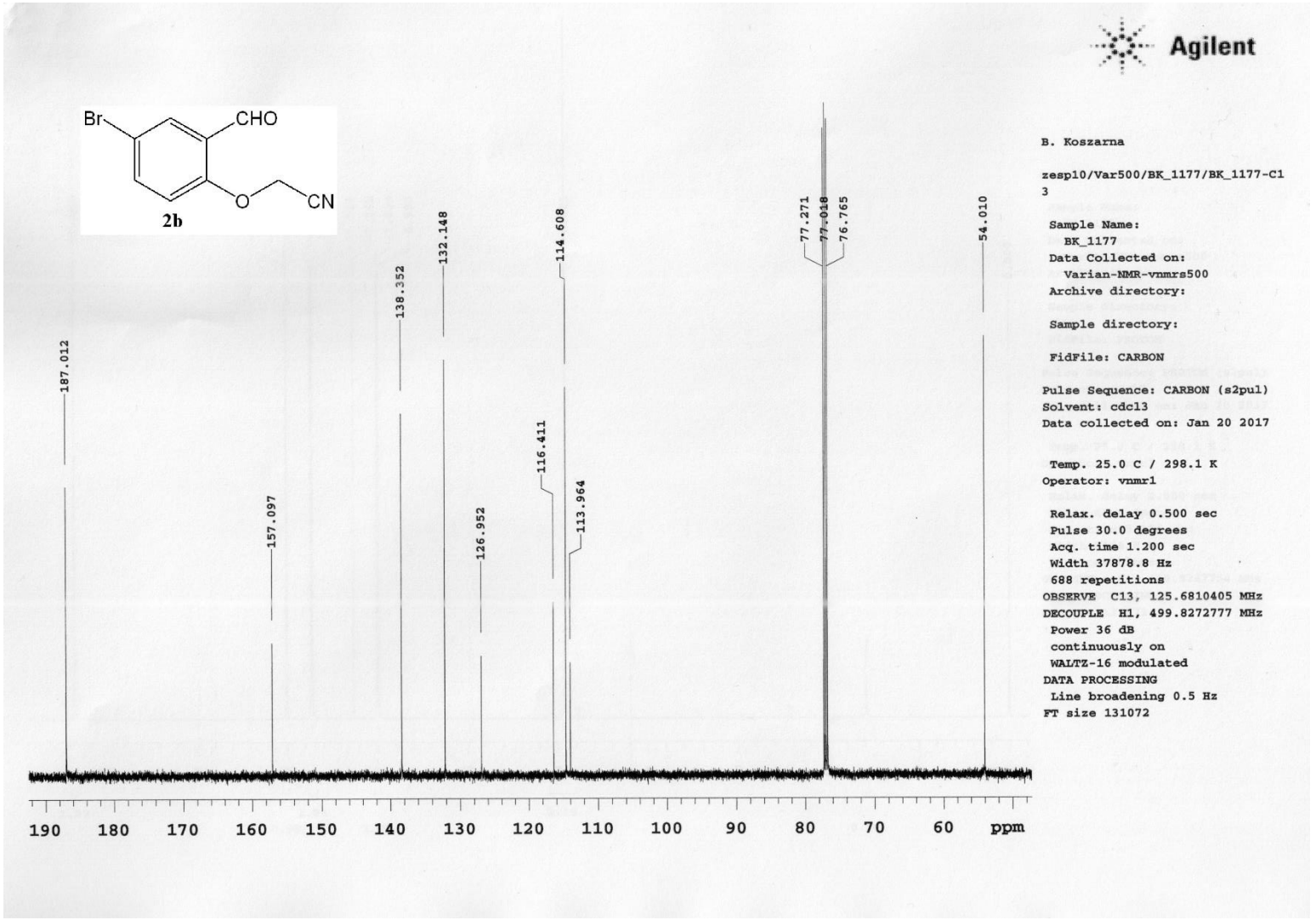
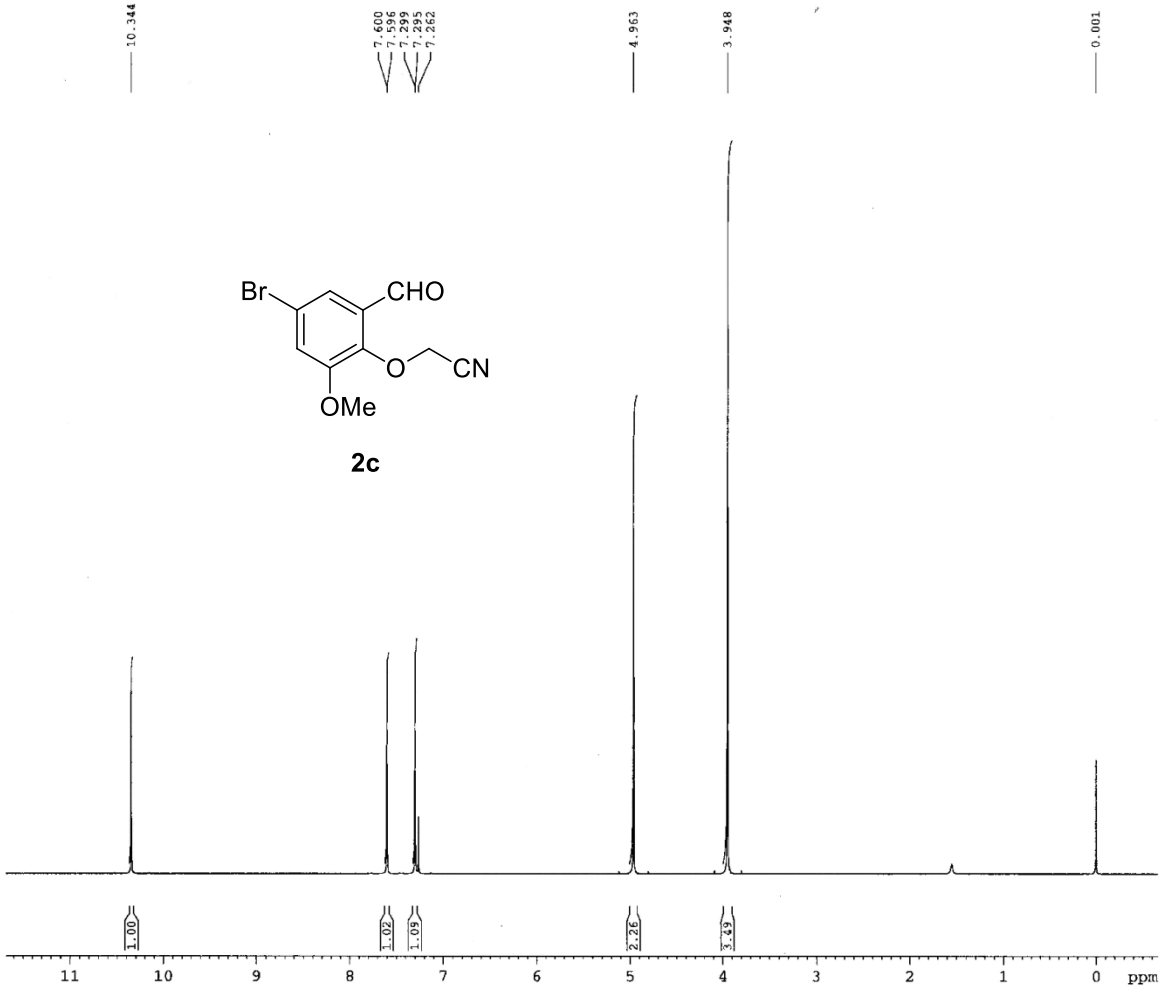
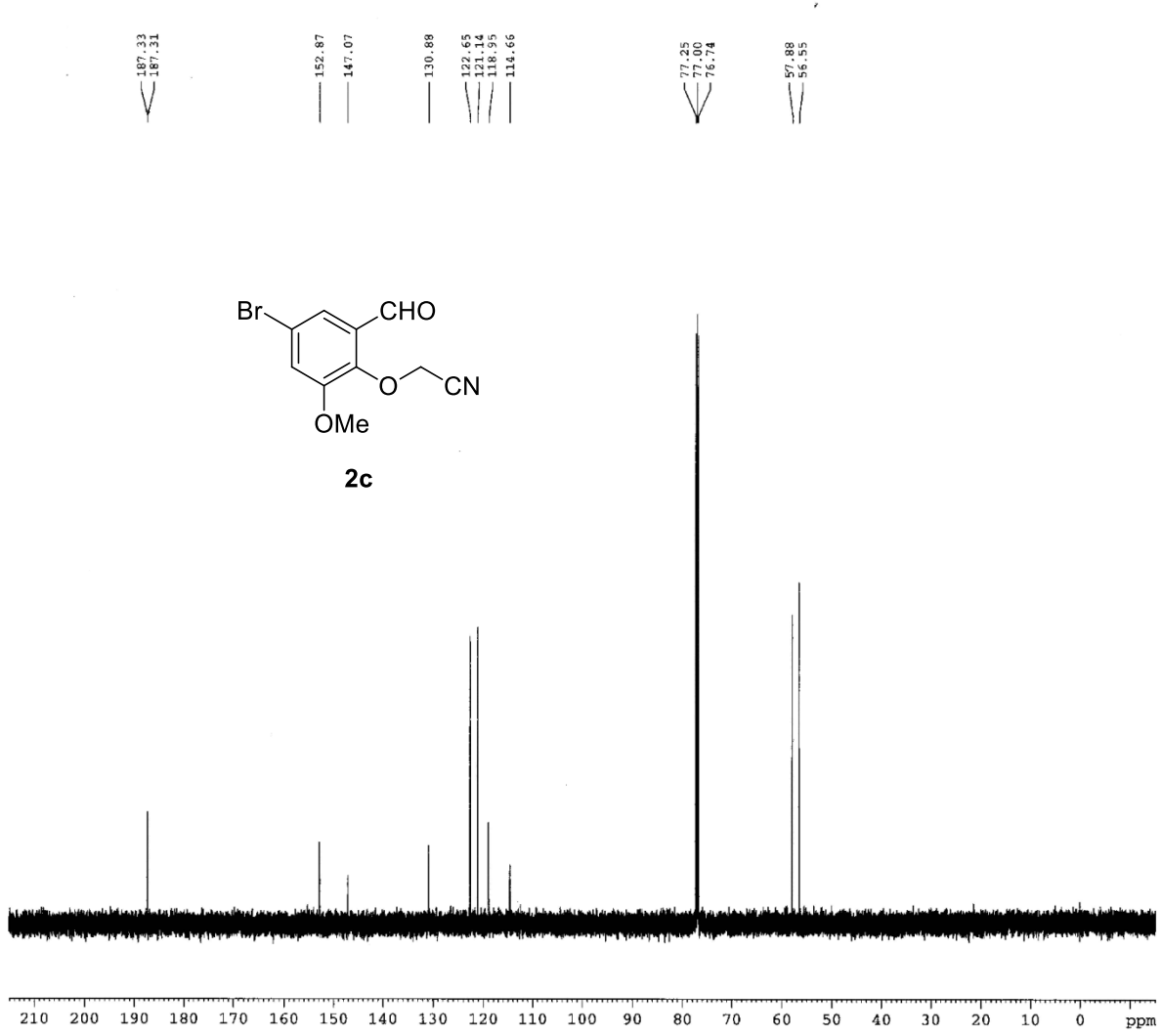
DATA PROCESSING
FT size 131072


Agilent
Br
CHO
O
CN
2b
187.012
157.097
138.352
132.148
126.952
116.411
114.608
113.964
77.271
77.018
76.765
54.010
190
180
170
160
150
140
130
120
110
100
90
80
70
60
ppm
B. Koszarna
zesp10/Var500/BK_1177/BK_1177-C13
Sample Name:
BK_1177
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 0.500 sec
Pulse 30.0 degrees
Acq. time 1.200 sec
Width 37878.8 Hz
688 repetitions
OBSERVE C13, 125.6810405 MHz
DECOUPLE H1, 499.8272777 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072



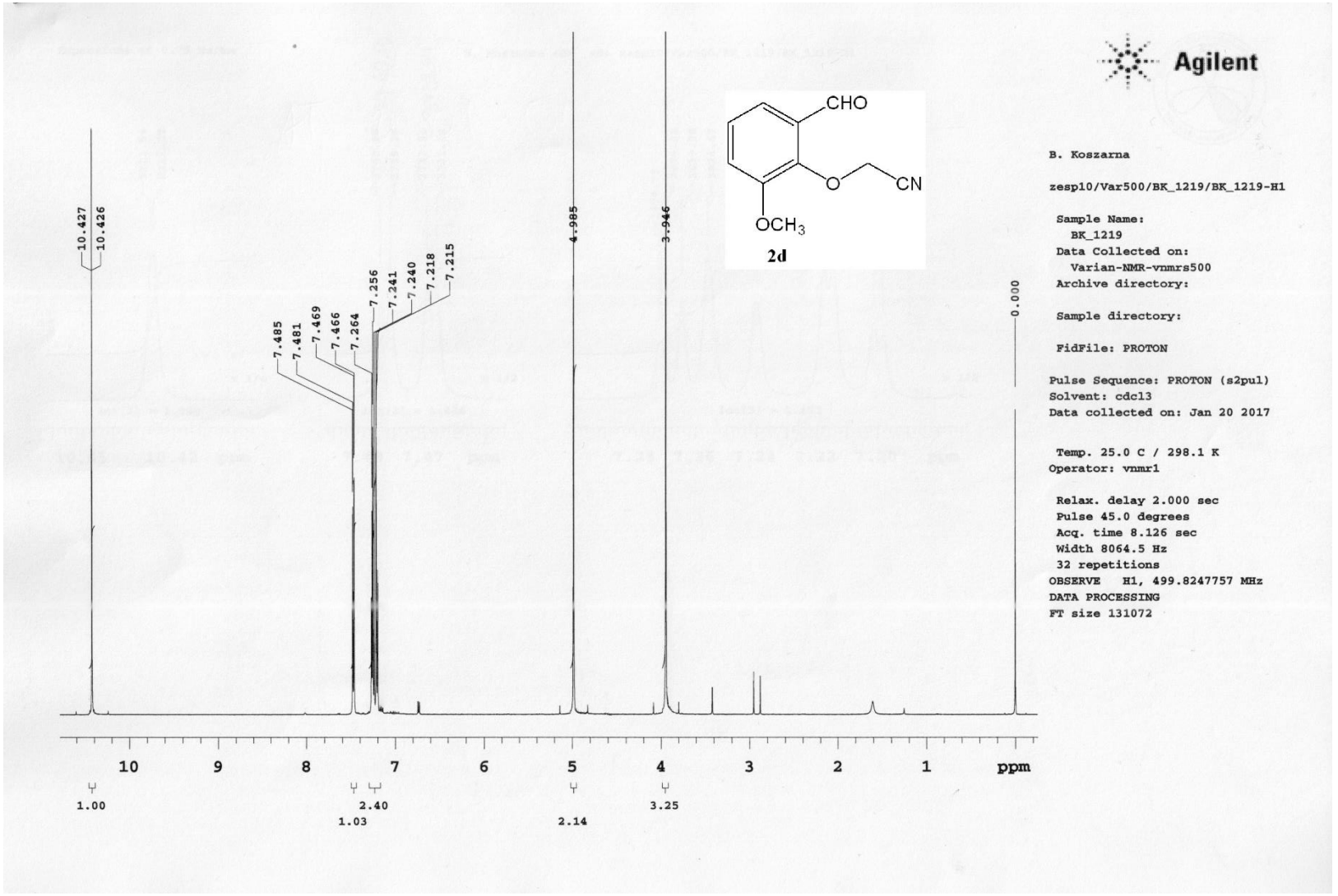
BRUKER

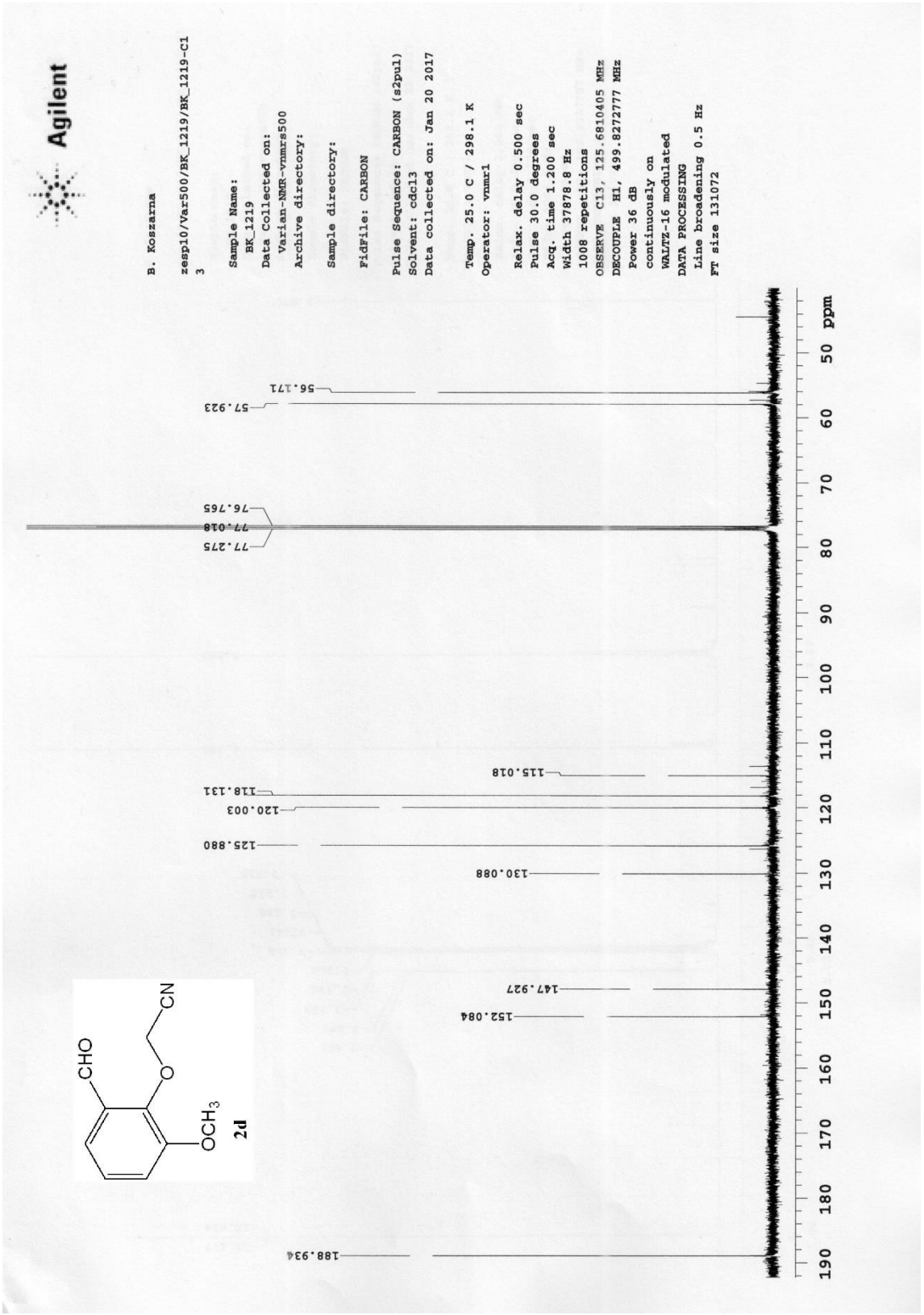
```
Current Data Parameters
NAME          BK-1180-new
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              20160104
Time                  12.13
INSTRUM                 DRX
PROBHD    5 mm TBI 1H/13
PULPROG                 zg
TD                   65536
SOLVENT              CDCl3
NS                      32
DS                       0
SWH              10330.578 Hz
FIDRES           0.157632 Hz
AQ               3.1719923 sec
RG                     256
DW                  48.400 usec
DE                   6.78 usec
TE                  303.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  2.50 usec
PL1                 0.00 dB
SFO1         500.1330885 MHz

F2 - Processing parameters
SI                  32768
SF           500.1300225 MHz
WDW                    no
SSB                     0
LB                  0.00 Hz
GB                      0
PC                   1.00
```

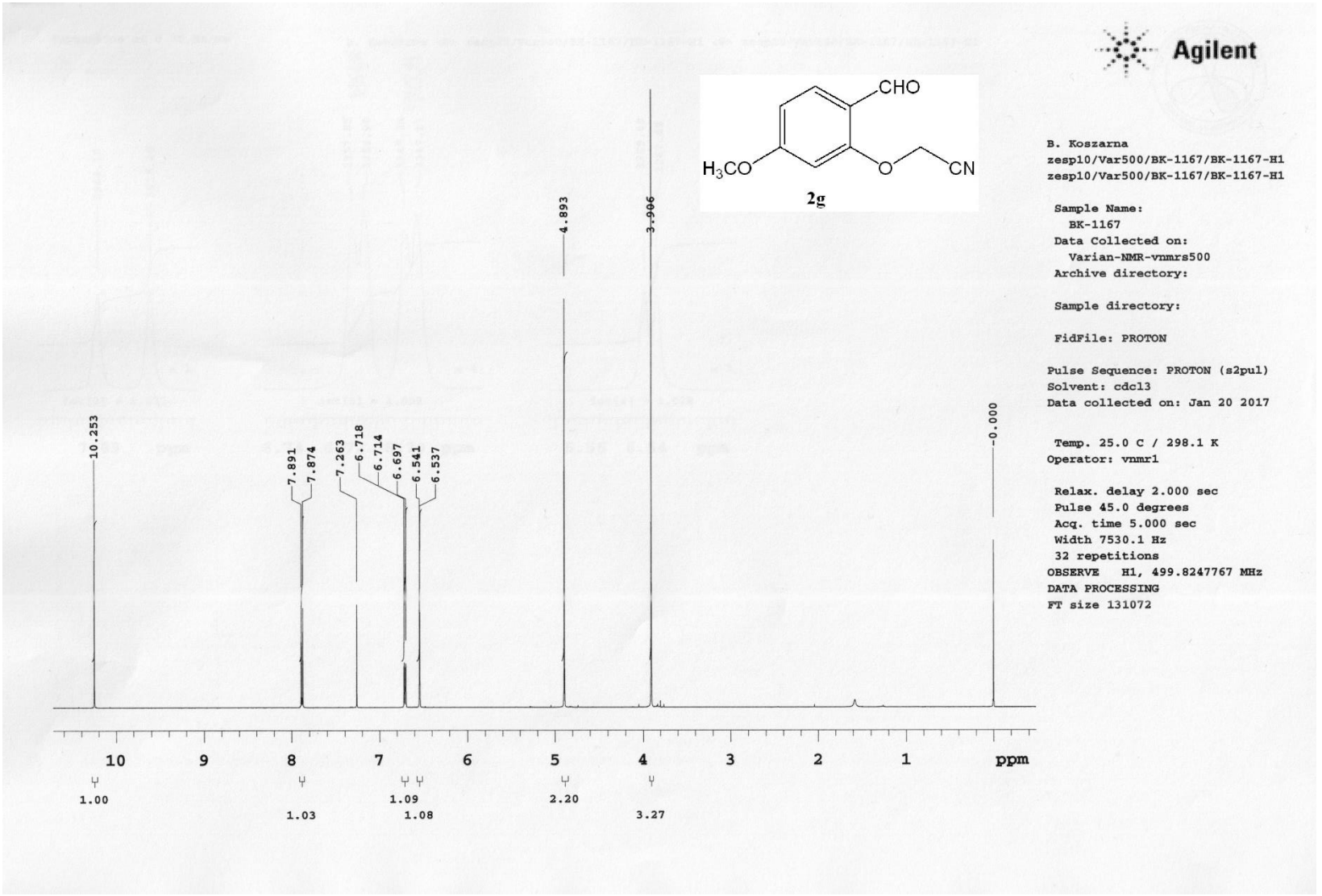


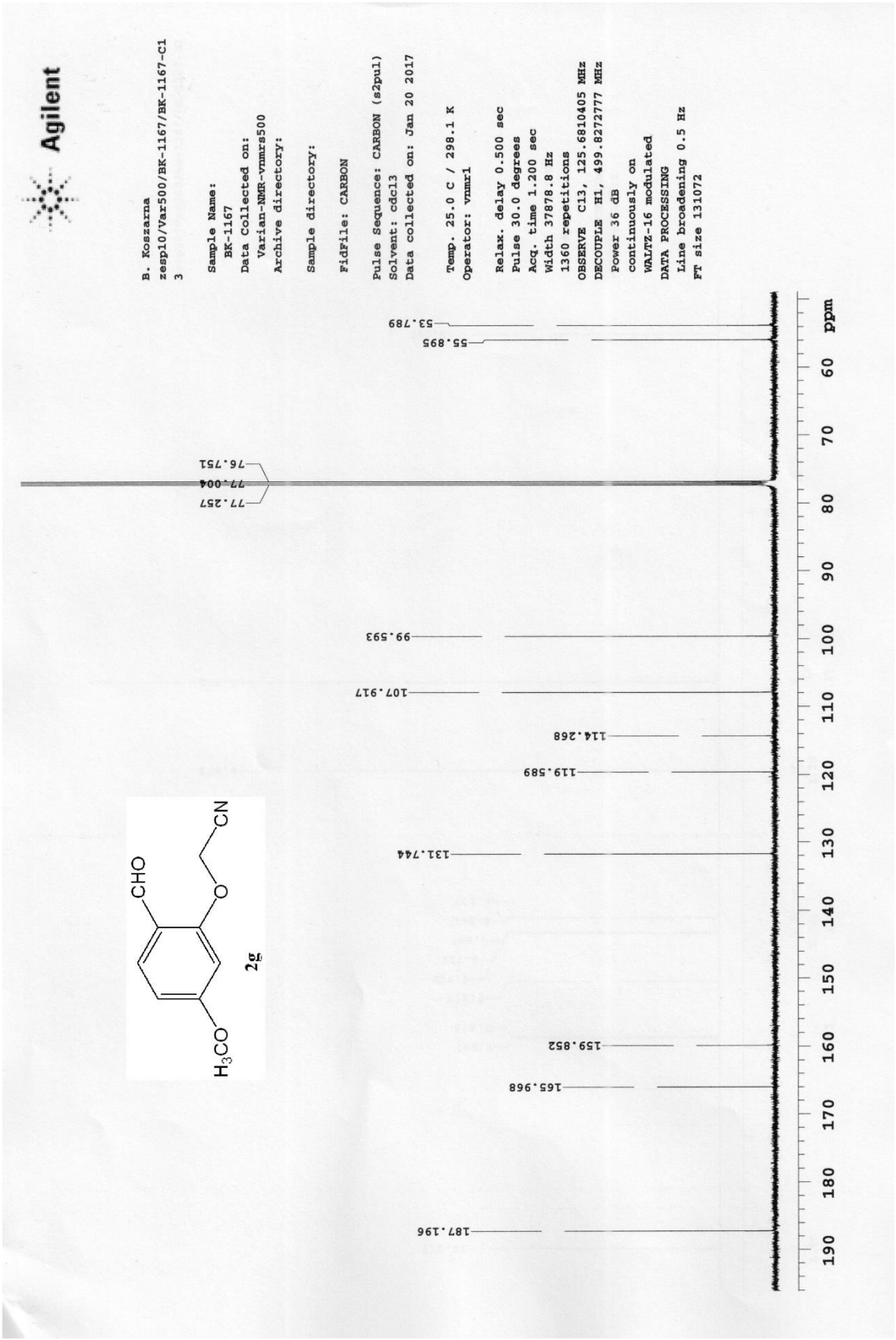
BRUKER

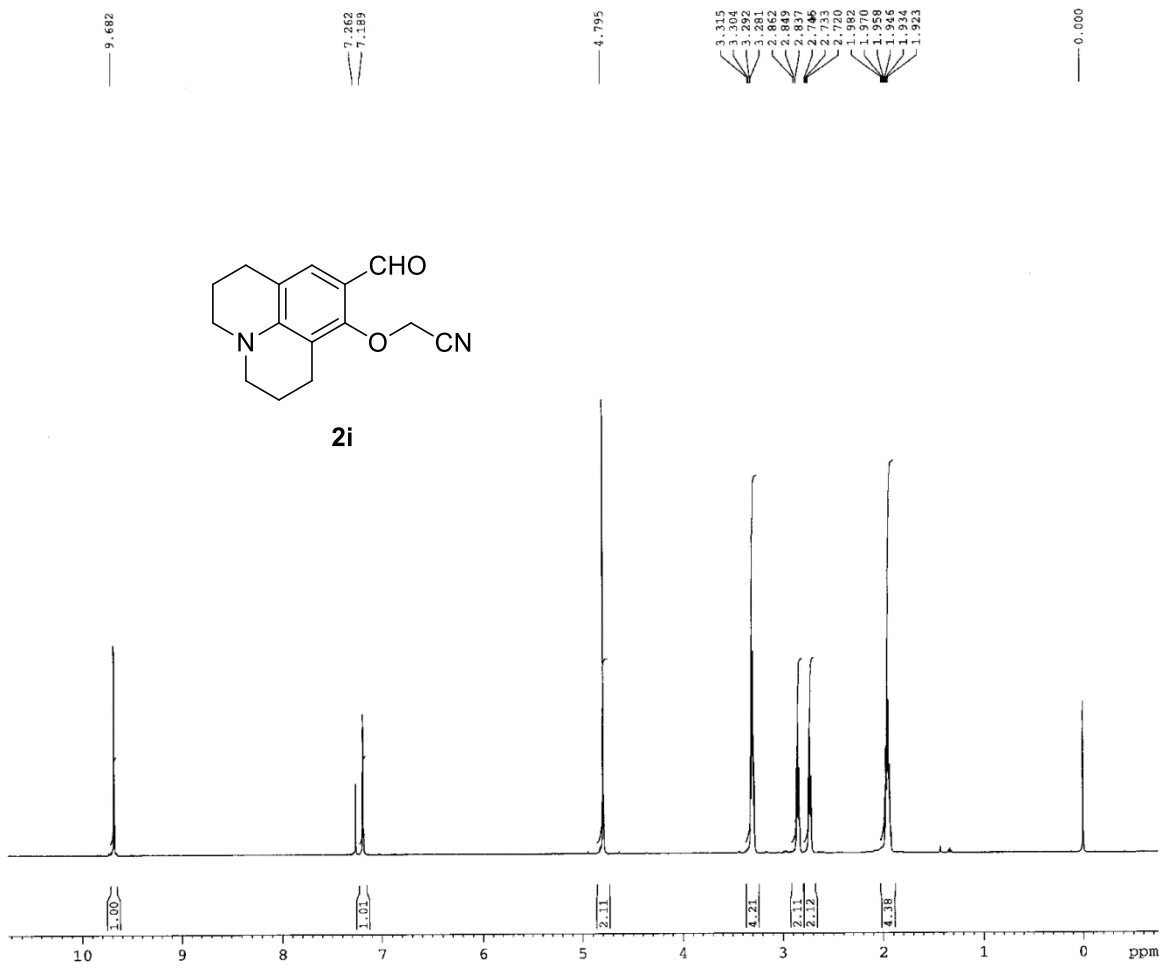
```
Current Data Parameters
NAME          BK-1180-new
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160104
Time                12.50
INSTRUM               DRX
PROBHD   5 mm TBI 1H/13
PULPROG               zgpg
TD                   65536
SOLVENT             CDCl3
NS                    1000
DS                       4
SWH            32679.738 Hz
FIDRES          0.498653 Hz
AQ              1.0027508 sec
RG                   32768
DW                15.300 usec
DE                 7.10 usec
TE                303.0 K
D1             1.00000000 sec
d11            0.03000000 sec
DELTA          0.89999998 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                  5.00 usec
PL1                -3.00 dB
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              98.00 usec
PL2                 3.00 dB
PL12               23.00 dB
PL13               32.00 dB
SFO2         500.1320005 MHz

F1 - Acquisition parameters
ND0                      1
TD                     128
SFO1           500.132 MHz
FIDRES         7.812500 Hz
SW                 1.999 ppm
FnMODE                 QF

F2 - Processing parameters
SI                  262144
SF           125.7577936 MHz
WDW                    EM
SSB                     0
LB                   0.50 Hz
GB                      0
PC                   1.40

F1 - Processing parameters
SI                    1024
MC2                     QF
SF           500.1300000 MHz
WDW                  SINE
SSB                     0
LB                   0.30 Hz
GB                    0.1
```


Agilent
CHO
O
CN
OCH3
2d
10.427
10.426
7.485
7.481
7.469
7.466
7.264
7.256
7.241
7.240
7.218
7.215
4.985
3.946
0.000
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.40
1.03
2.14
3.25
B. Koszarna
zesp10/Var500/BK_1219/BK_1219-H1
Sample Name:
BK_1219
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 8.126 sec
Width 8064.5 Hz
32 repetitions
OBSERVE H1, 499.8247757 MHz
DATA PROCESSING
FT size 131072


Agilent
B. Koszarna
zesp10/Var500/BK_1219/BK_1219-C1
3
Sample Name:
BK_1219
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 0.500 sec
Pulse 30.0 degrees
Acq. time 1.200 sec
Width 37878.8 Hz
1008 repetitions
OBSERVE C13, 125.6810405 MHz
DECOUPLE H1, 499.8272777 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
56.171
57.923
76.765
77.018
77.275
115.018
118.131
120.003
125.880
130.088
147.927
152.084
188.934
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 ppm
CHO
O
CN
OCH3
2d

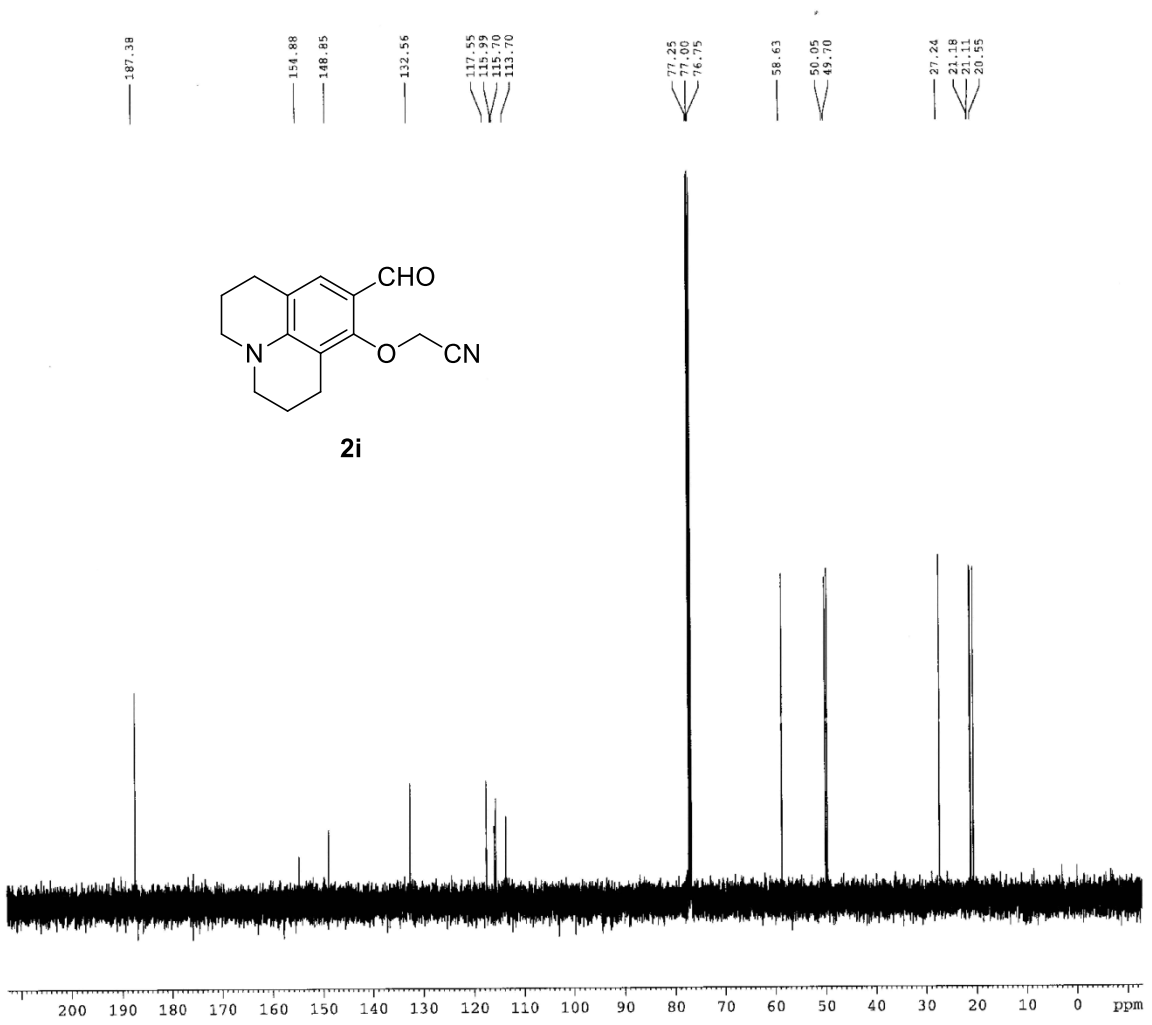

Agilent
CHO
H_3CO
O
CN
2g
10.253
7.891
7.874
7.263
6.718
6.714
6.697
6.541
6.537
4.893
3.906
0.000
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.03
1.09
1.08
2.20
3.27
B. Koszarna
zesp10/Var500/BK-1167/BK-1167-H1
zesp10/Var500/BK-1167/BK-1167-H1
Sample Name:
BK-1167
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 5.000 sec
Width 7530.1 Hz
32 repetitions
OBSERVE H1, 499.8247767 MHz
DATA PROCESSING
FT size 131072


Agilent
B. Koszarna
zesp10/Var500/BK-1167/BK-1167-C1
3
Sample Name:
BK-1167
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 0.500 sec
Pulse 30.0 degrees
Acq. time 1.200 sec
Width 37878.8 Hz
1360 repetitions
OBSERVE C13, 125.6810405 MHz
DECOUPLE H1, 499.8272777 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
53.789
55.895
76.751
77.004
77.257
99.593
107.917
114.268
119.589
131.744
159.852
165.968
187.196
ppm
CHO
CN
O
H3CO
2g



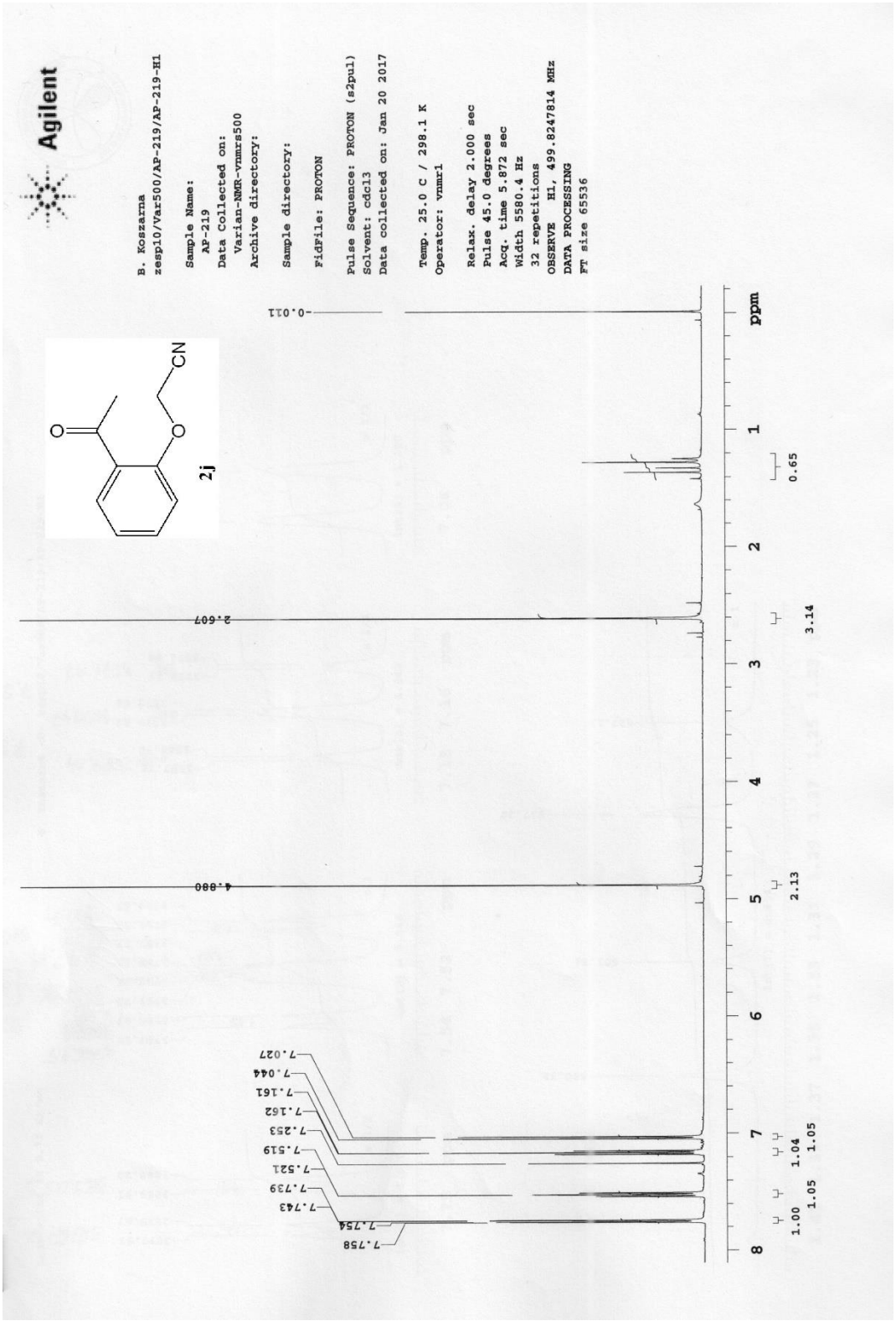
BRUKER

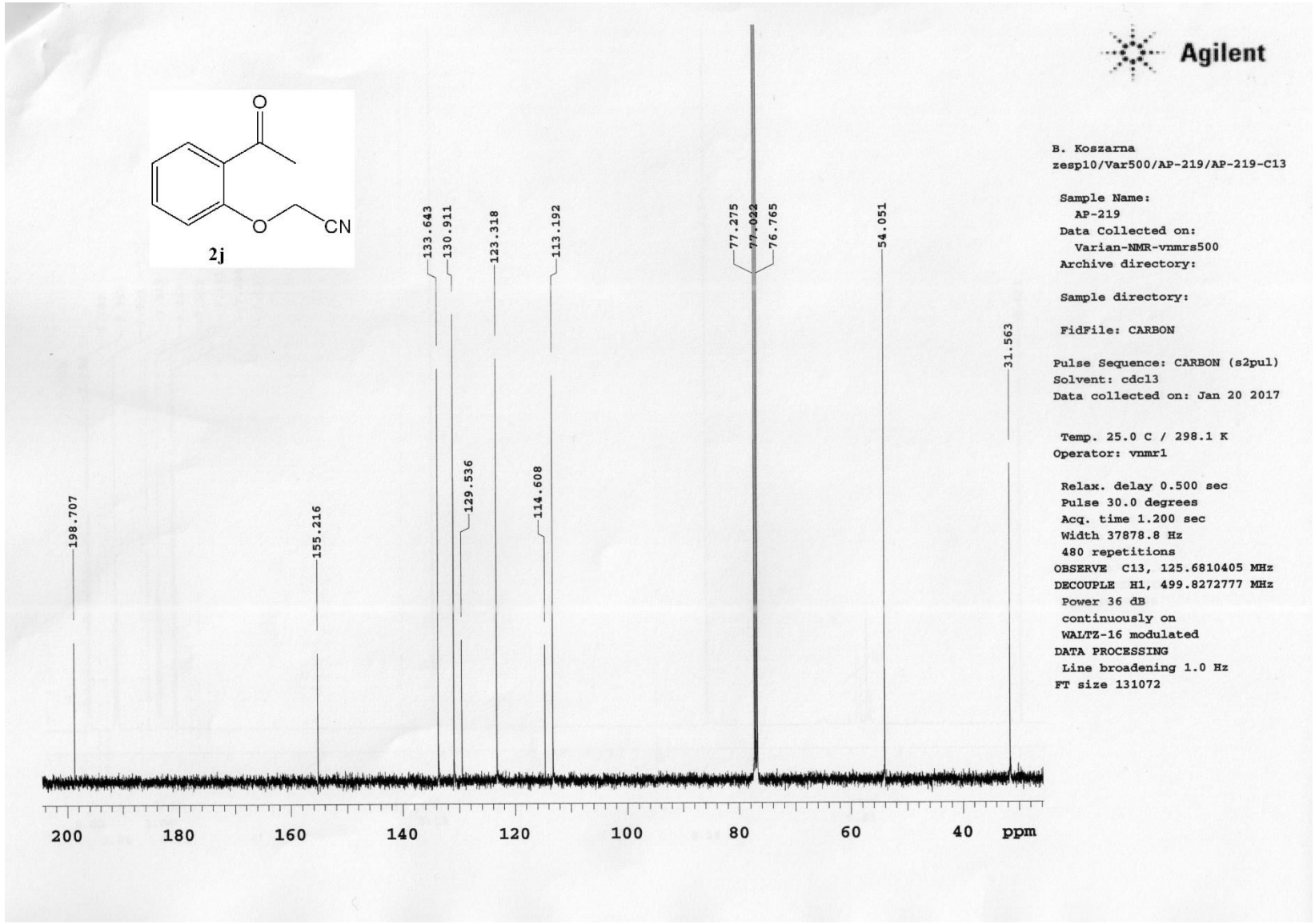
```
Current Data Parameters
NAME                  AP236
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20151218
Time                  12.39
INSTRUM                DRX
PROBHD   5 mm TBI 1H/13
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       32
DS                        0
SWH               10330.578 Hz
FIDRES            0.157632 Hz
AQ                3.1719923 sec
RG                      181
DW                 48.400 usec
DE                  6.78 usec
TE                  303.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                  8.20 usec
PL1                 5.00 dB
SFO1          500.1330885 MHz

F2 - Processing parameters
SI                    32768
SF            500.1300230 MHz
WDW                      no
SSB                       0
LB                  0.00 Hz
GB                        0
PC                     1.00
```

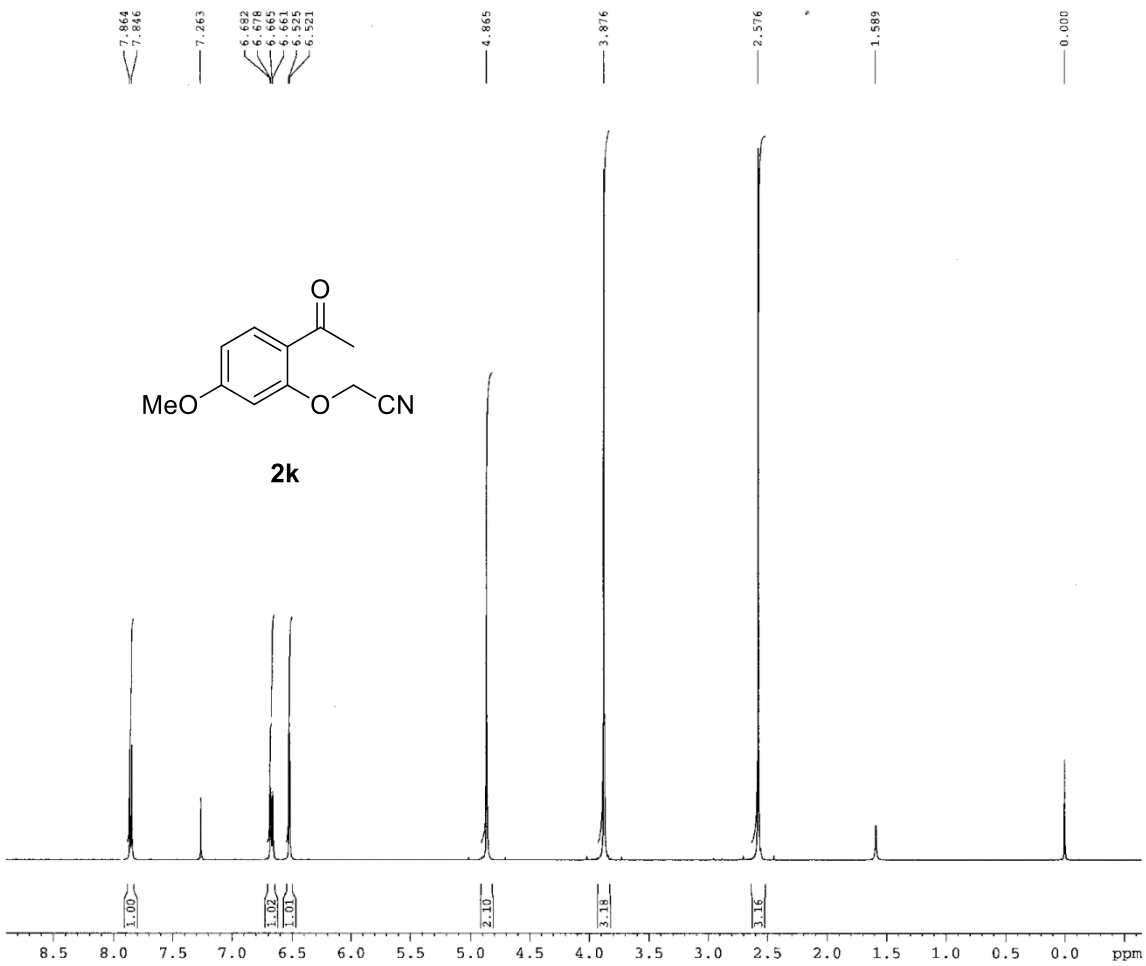


BRUKER

```
Current Data Parameters
NAME                  AP236
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20151218
Time                  12.45
INSTRUM                 DRX
PROBHD   5 mm TBI 1H/13
PULPROG                zgpg
TD                    65536
SOLVENT               CDCl3
NS                      550
DS                        4
SWH               32679.738 Hz
FIDRES            0.498653 Hz
AQ                1.0027508 sec
RG                    32768
DW                   15.300 usec
DE                    7.10 usec
TE                   303.0 K
D1                1.00000000 sec
d11               0.03000000 sec
DELTA             0.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     5.00 usec
PL1                   -3.00 dB
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 98.00 usec
PL2                    3.00 dB
PL12                  23.00 dB
PL13                  32.00 dB
SFO2           500.1320005 MHz

F1 - Acquisition parameters
ND0                       1
TD                      128
SFO1               500.132 MHz
FIDRES            7.812500 Hz
SW                    1.999 ppm
FnMODE                   QF

F2 - Processing parameters
SI                   262144
SF             125.7577943 MHz
WDW                      EM
SSB                       0
LB                     0.50 Hz
GB                        0
PC                     1.40

F1 - Processing parameters
SI                     1024
MC2                      QF
SF             500.1300000 MHz
WDW                    SINE
SSB                       0
LB                     0.30 Hz
GB                      0.1
```


Agilent
B. Koszarna
zesp10/Var500/AP-219/AP-219-H1
Sample Name:
AP-219
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 5.872 sec
Width 5580.4 Hz
32 repetitions
OBSERVE H1, 499.8247814 MHz
DATA PROCESSING
FT size 65536
CN
O
O
2j
-0.011
2.607
4.880
7.027
7.044
7.161
7.162
7.253
7.519
7.521
7.739
7.743
7.754
7.758
ppm
1.00
1.04
1.05
1.05
2.13
3.14
0.65


Agilent
O
O
CN
2j
198.707
155.216
133.643
130.911
129.536
123.318
114.608
113.192
77.275
77.022
76.765
54.051
31.563
200
180
160
140
120
100
80
60
40
ppm
B. Koszarna
zesp10/Var500/AP-219/AP-219-C13
Sample Name:
AP-219
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 0.500 sec
Pulse 30.0 degrees
Acq. time 1.200 sec
Width 37878.8 Hz
480 repetitions
OBSERVE C13, 125.6810405 MHz
DECOUPLE H1, 499.8272777 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072



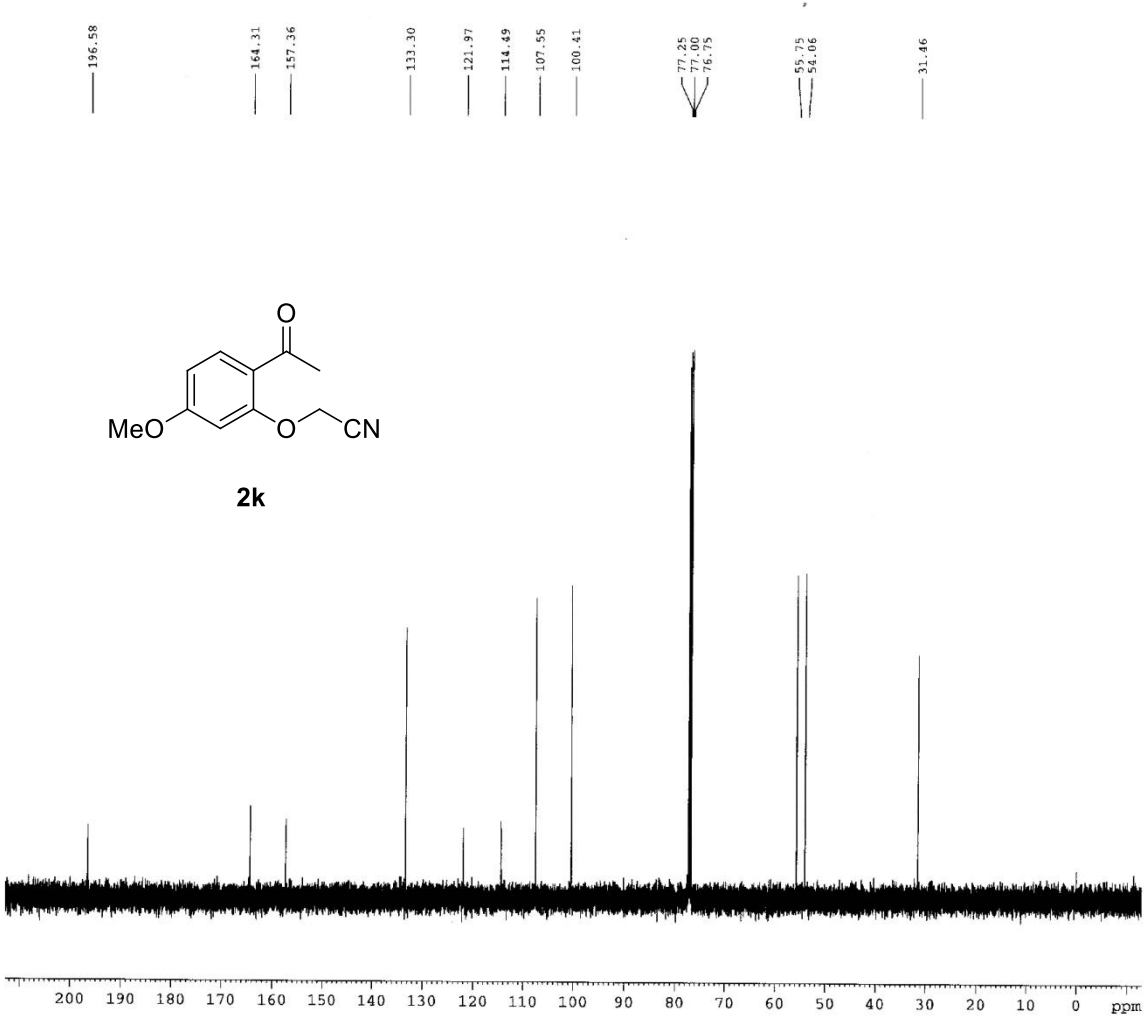
BRUKER

Current Data Parameters
NAME AP245
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151119
Time 17.08
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 228.1
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

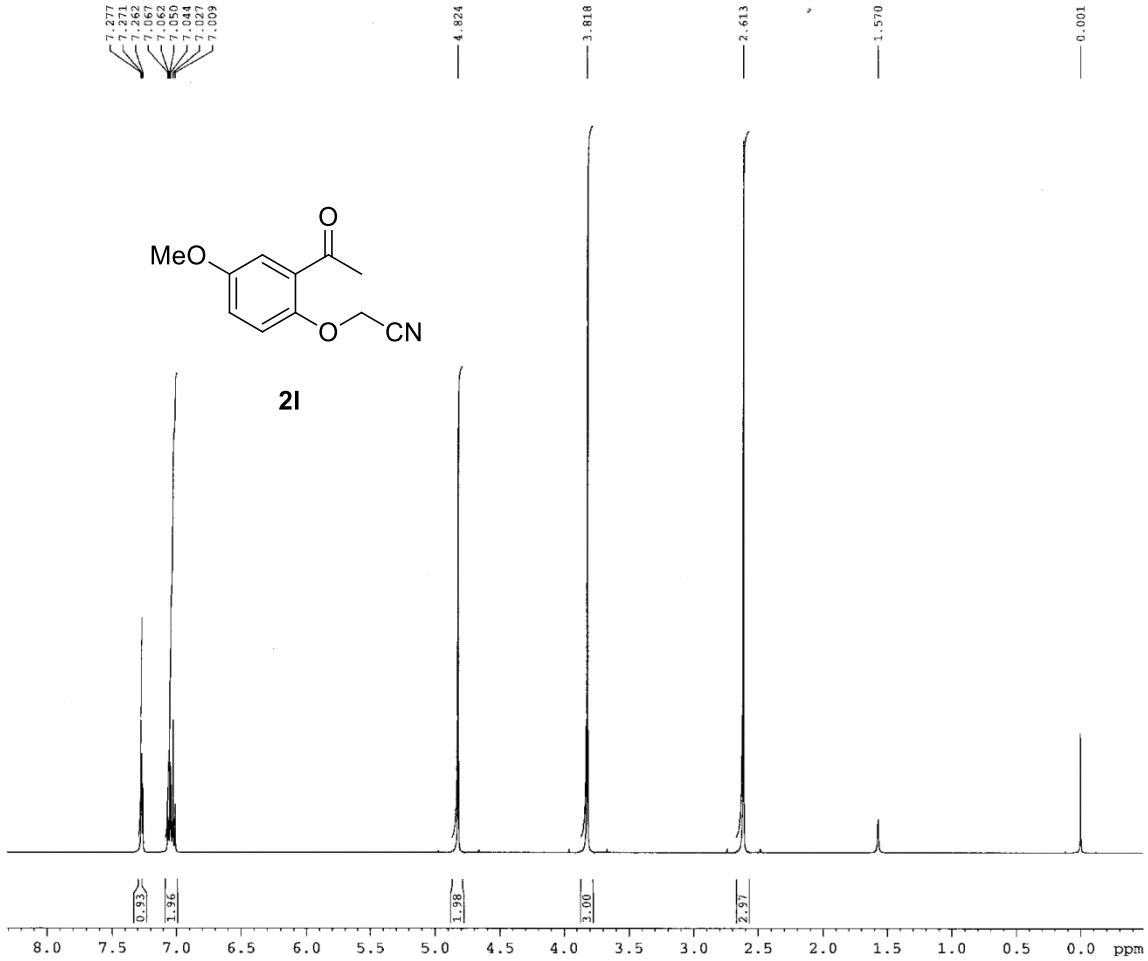
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300220 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



BRUKER

```
Current Data Parameters
NAME                  AP245
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20151119
Time                  17.22
INSTRUM                 DRX
PROBHD    5 mm TBI 1H/13
PULPROG                zgpg
TD                    65536
SOLVENT               CDCl3
NS                      500
DS                        4
SWH               32679.738 Hz
FIDRES            0.498653 Hz
AQ                1.0027508 sec
RG                    32768
DW                 15.300 usec
DE                  7.10 usec
TE                  303.0 K
D1              1.00000000 sec
d11             0.03000000 sec
DELTA           0.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                   5.00 usec
PL1                 -3.00 dB
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2               98.00 usec
PL2                  3.00 dB
PL12                23.00 dB
PL13                32.00 dB
SFO2          500.1320005 MHz

F1 - Acquisition parameters
ND0                       1
TD                      128
SFO1            500.132 MHz
FIDRES          7.812500 Hz
SW                  1.999 ppm
FnMODE                 QF

F2 - Processing parameters
SI                   262144
SF            125.7577940 MHz
WDW                    EM
SSB                     0
LB                   0.50 Hz
GB                      0
PC                   1.40

F1 - Processing parameters
SI                     1024
MC2                    QF
SF            500.1300000 MHz
WDW                  SINE
SSB                     0
LB                   0.30 Hz
GB                    0.1
```



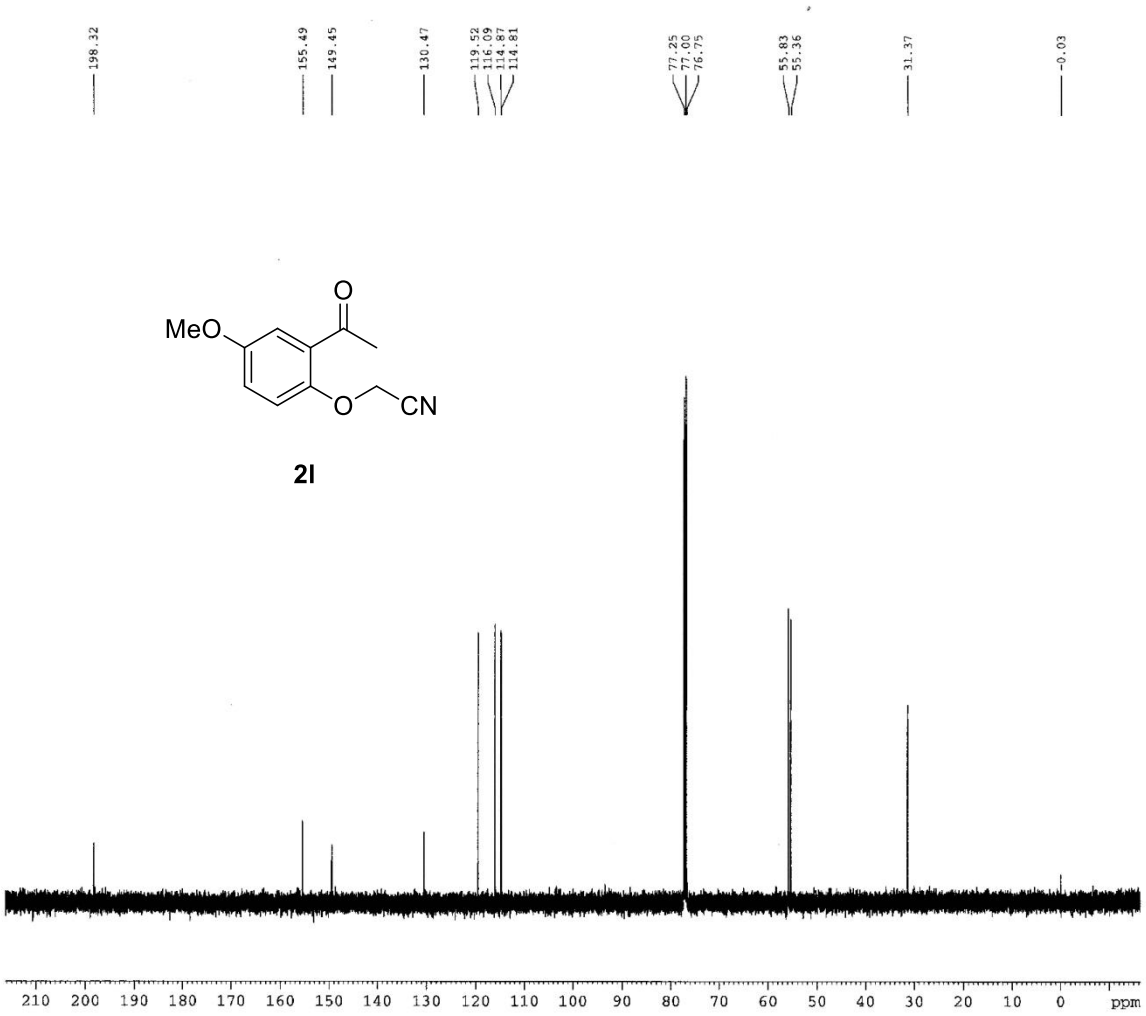
BRUKER

Current Data Parameters
NAME AP260
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151218
Time 13.08
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 287.4
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300227 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



BRUKER

Current Data Parameters
NAME AP260
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151218
Time 13.41
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1000
DS 4
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027508 sec
RG 32768
DW 15.300 usec
DE 7.10 usec
TE 303.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

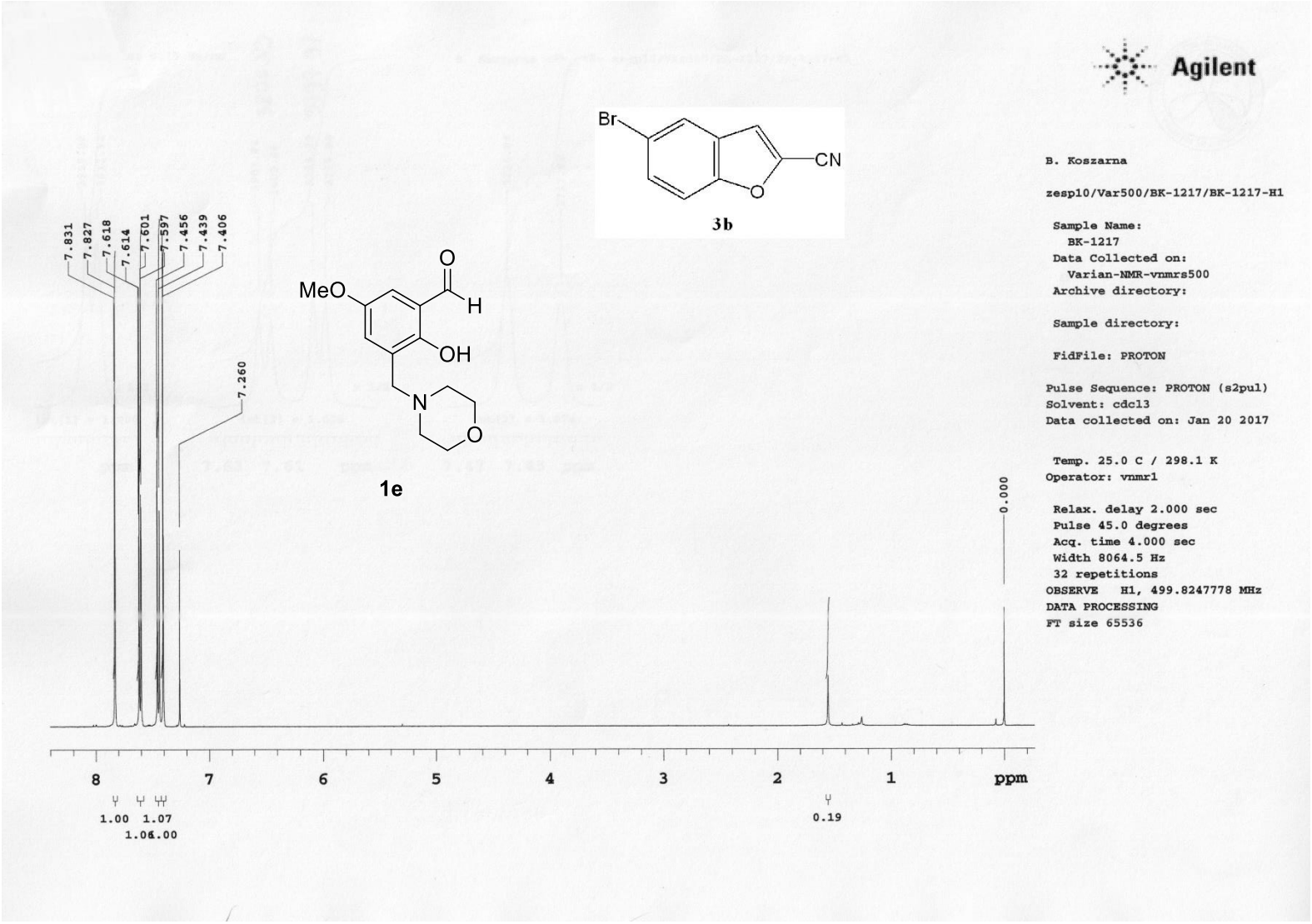
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -3.00 dB
SFO1 125.7703643 MHz

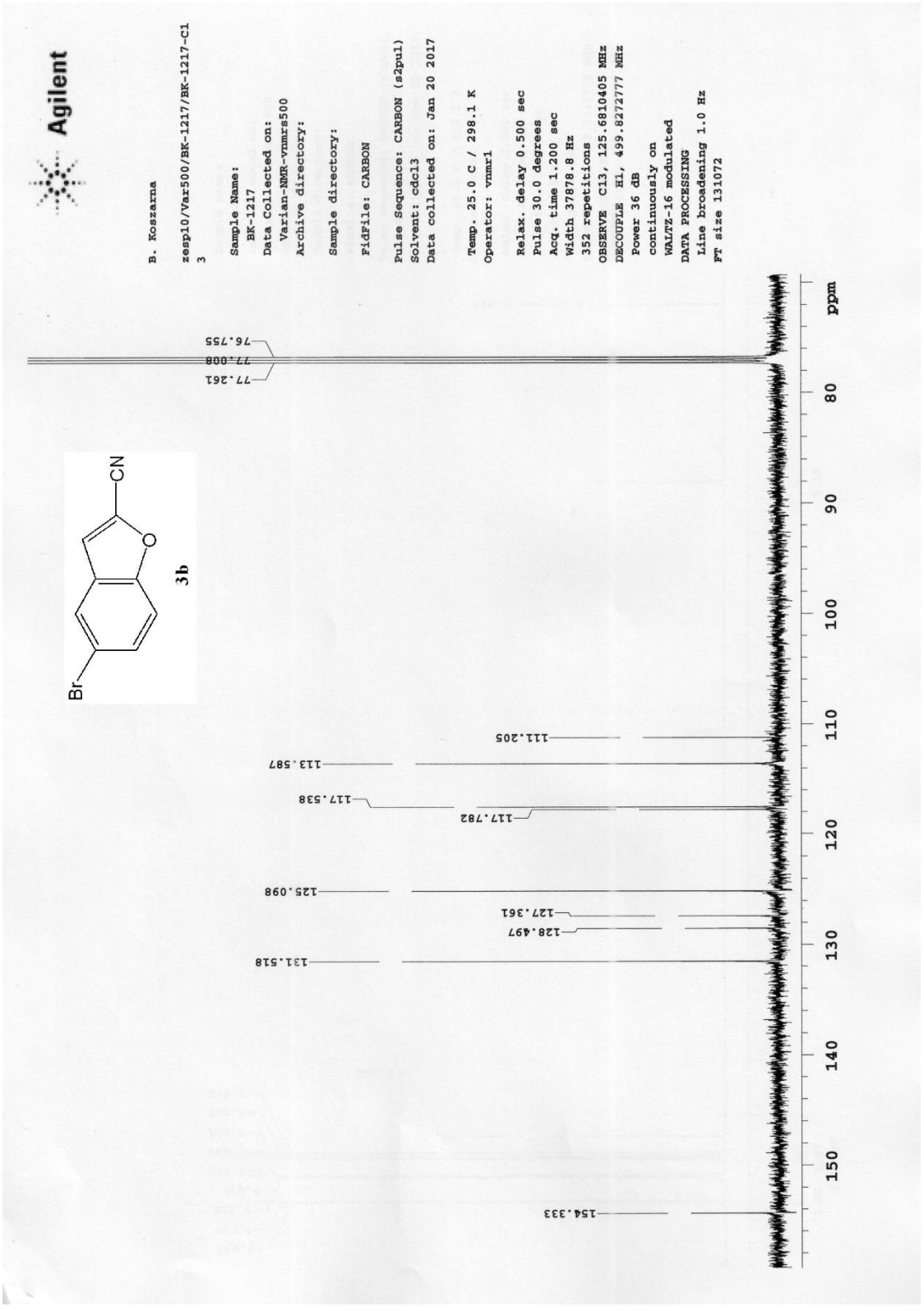
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.00 usec
PL2 3.00 dB
PL12 23.00 dB
PL13 32.00 dB
SFO2 500.1320005 MHz

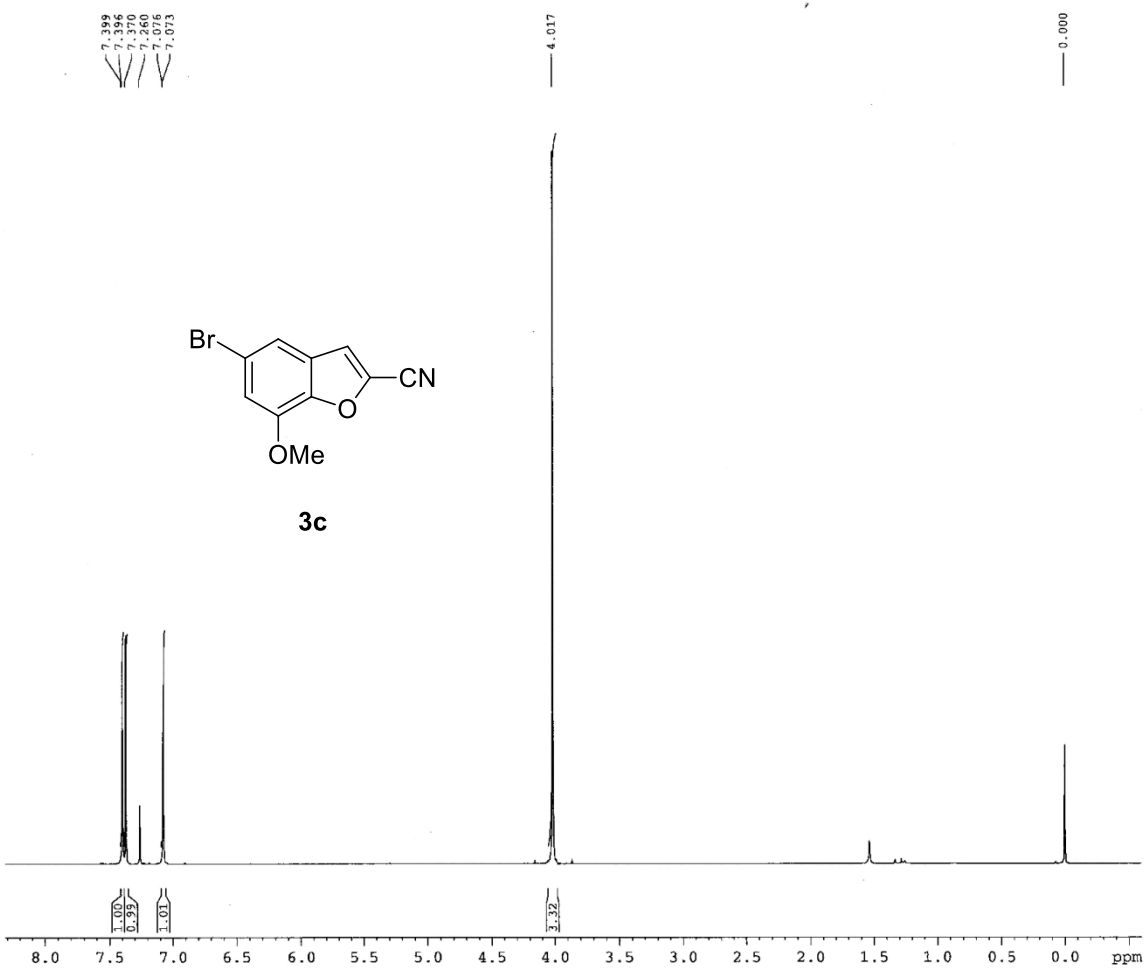
F1 - Acquisition parameters
ND0 1
TD 128
SFO1 500.132 MHz
FIDRES 7.812500 Hz
SW 1.999 ppm
FnMODE QF

F2 - Processing parameters
SI 262144
SF 125.7577940 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0.30 Hz
GB 0.1


Agilent
B. Koszarna
zesp10/Var500/BK-1217/BK-1217-H1
Sample Name:
BK-1217
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 8064.5 Hz
32 repetitions
OBSERVE H1, 499.8247778 MHz
DATA PROCESSING
FT size 65536
Br
CN
O
3b
MeO
O
H
OH
N
O
1e
7.831
7.827
7.618
7.614
7.601
7.597
7.456
7.439
7.406
7.260
0.000
8
7
6
5
4
3
2
1
ppm
1.00
1.07
1.06
4.00
0.19


Agilent
B. Koszarna
zesp10/Var500/BK-1217/BK-1217-C1
3
Sample Name:
BK-1217
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 0.500 sec
Pulse 30.0 degrees
Acq. time 1.200 sec
Width 37878.8 Hz
352 repetitions
OBSERVE C13, 125.6810405 MHz
DECOUPLE H1, 499.8272777 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
77.261
77.008
76.755
111.205
113.587
117.538
117.782
125.098
127.361
128.497
131.518
154.333
ppm
CN
O
Br
3b



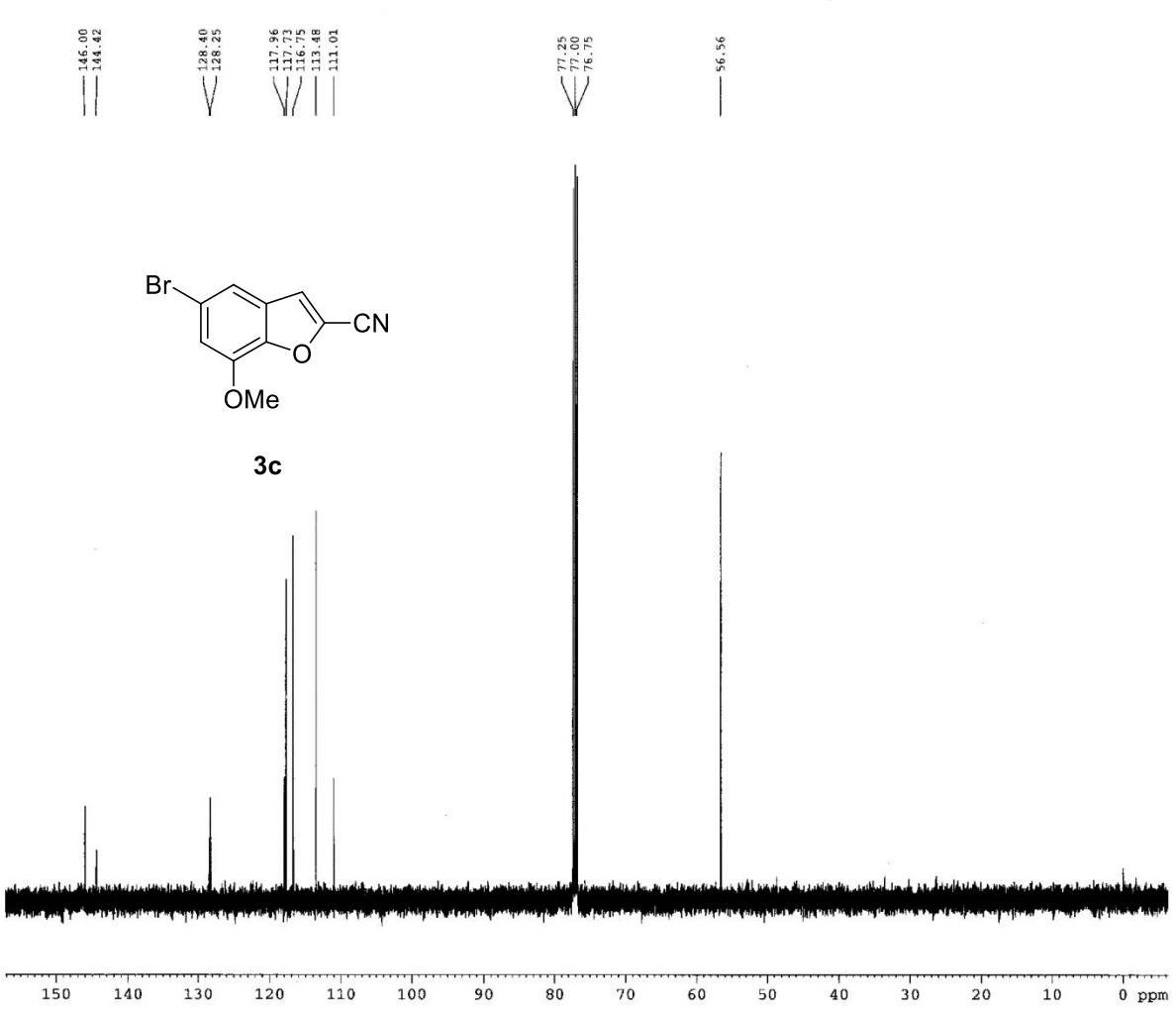
BRUKER

Current Data Parameters
NAME BK-1224
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160104
Time 10.56
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 287.4
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

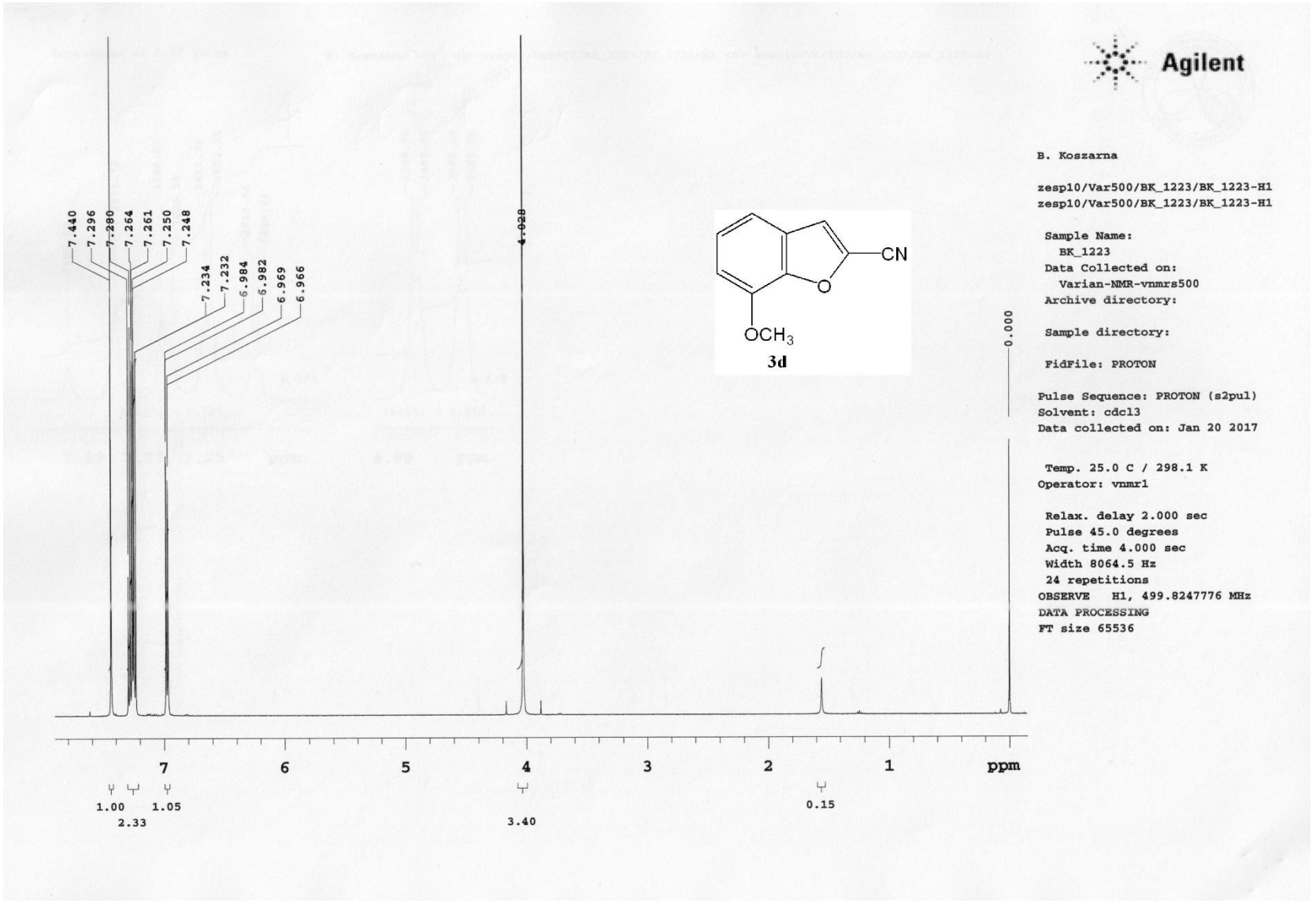
======== CHANNEL f1 ========
NUC1 1H
P1 2.50 usec
PL1 0.00 dB
SFO1 500.1330885 MHz

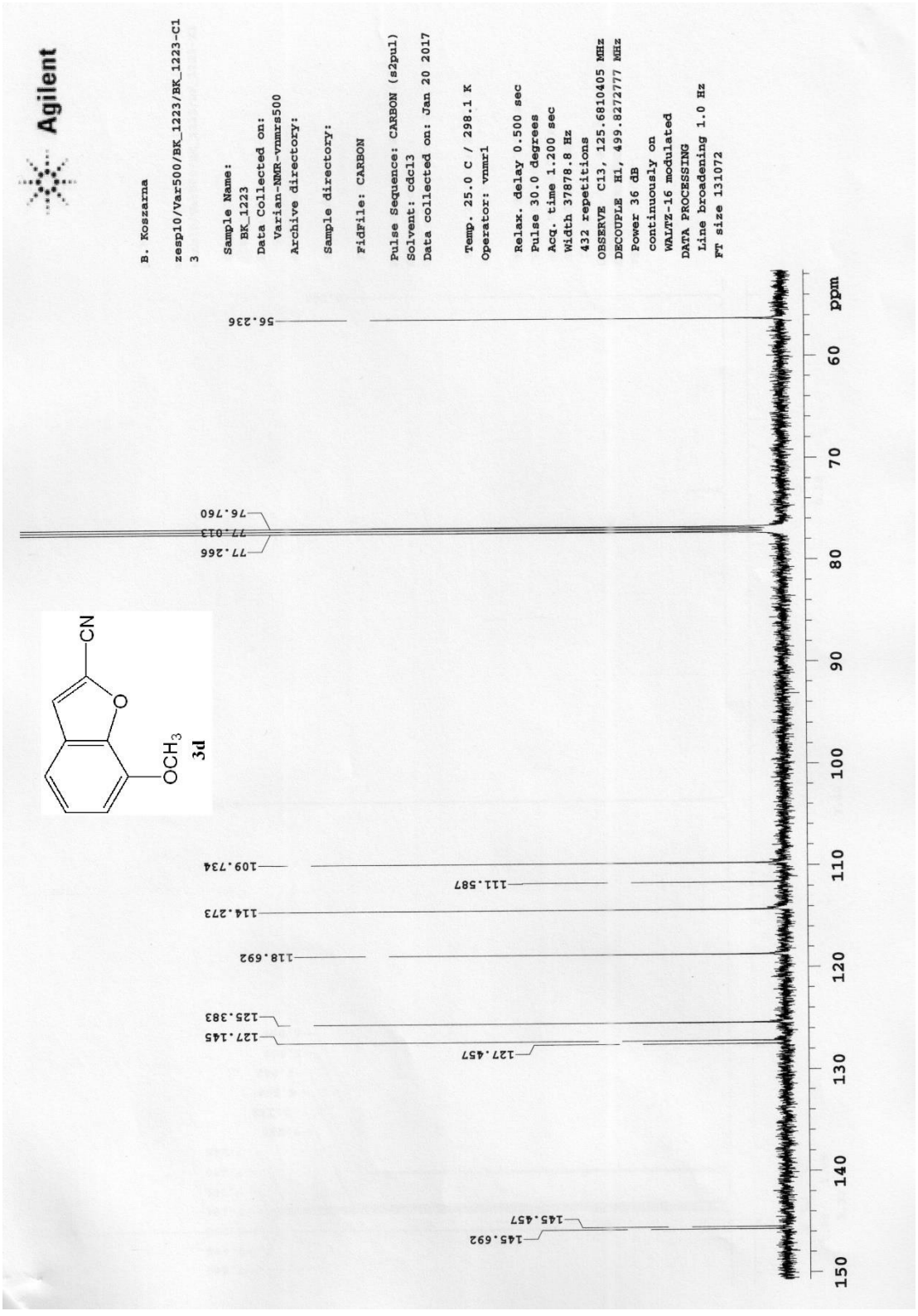
F2 - Processing parameters
SI 32768
SF 500.1300234 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

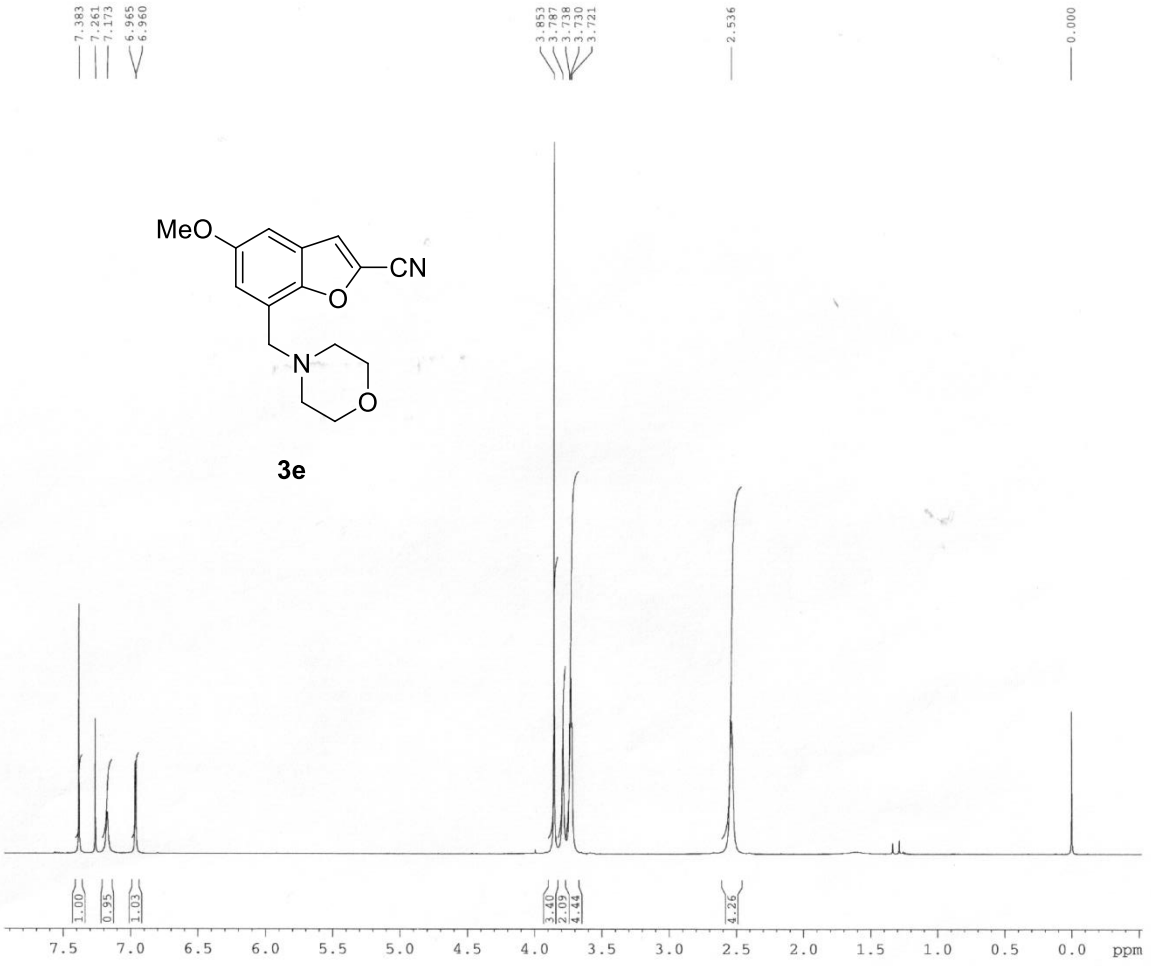


BRUKER

```
Current Data Parameters
NAME                  BK-1224
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_                20160104
Time                    11.32
INSTRUM                   DRX
PROBHD   5 mm TBI 1H/13
PULPROG                 zgpg
TD                     65536
SOLVENT                CDCl3
NS                      1000
DS                         4
SWH                32679.738 Hz
FIDRES             0.498653 Hz
AQ                 1.0027508 sec
RG                      32768
DW                   15.300 usec
DE                    7.10 usec
TE                    303.0 K
D1                 1.00000000 sec
d11                0.03000000 sec
DELTA              0.89999998 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                       5.00 usec
PL1                     -3.00 dB
SFO1              125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   98.00 usec
PL2                      3.00 dB
PL12                    23.00 dB
PL13                    32.00 dB
SFO2              500.1320005 MHz

F1 - Acquisition parameters
ND0                         1
TD                        128
SFO1                500.132 MHz
FIDRES             7.812500 Hz
SW                    1.999 ppm
FnMODE                     QF

F2 - Processing parameters
SI                     262144
SF                125.7577933 MHz
WDW                        EM
SSB                         0
LB                       0.50 Hz
GB                          0
PC                       1.40

F1 - Processing parameters
SI                       1024
MC2                        QF
SF                500.1300000 MHz
WDW                      SINE
SSB                         0
LB                       0.30 Hz
GB                        0.1
```


Agilent
B. Koszarna
zesp10/Var500/BK_1223/BK_1223-H1
zesp10/Var500/BK_1223/BK_1223-H1
Sample Name:
BK_1223
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 8064.5 Hz
24 repetitions
OBSERVE H1, 499.8247776 MHz
DATA PROCESSING
FT size 65536
7.440
7.296
7.280
7.264
7.261
7.250
7.248
7.234
7.232
6.984
6.982
6.969
6.966
4.028
0.000
CN
O
OCH3
3d
7
6
5
4
3
2
1
ppm
1.00
2.33
1.05
3.40
0.15


Agilent
B. Koszarna
zesp10/Var500/BK_1223/BK_1223-C1
3
Sample Name:
BK_1223
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 0.500 sec
Pulse 30.0 degrees
Acq. time 1.200 sec
Width 37878.8 Hz
432 repetitions
OBSERVE C13, 125.6810405 MHz
DECOUPLE H1, 499.8272777 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
56.236
76.760
77.013
77.266
109.734
111.587
114.273
118.692
125.383
127.145
127.457
145.457
145.692
ppm
150
140
130
120
110
100
90
80
70
60
CN
O
OCH3
3d



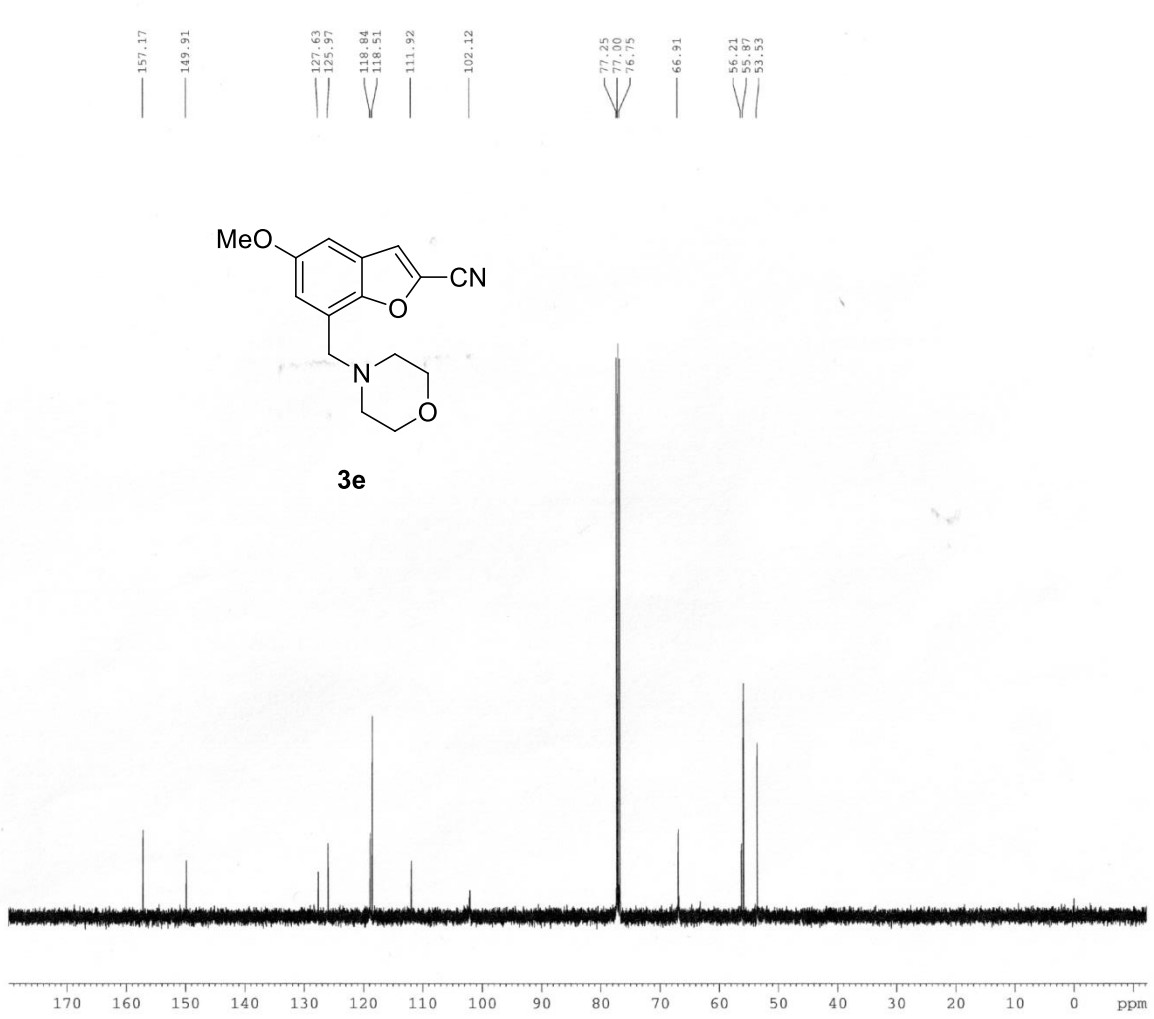
BRUKER

Current Data Parameters
NAME XY83
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150424
Time 12.00
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 161.3
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 2.50 usec
PL1 0.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300230 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



3e

BRUKER

Current Data Parameters
NAME XY83
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150424
Time 12.17
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1700
DS 4
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027508 sec
RG 32768
DW 15.300 usec
DE 7.10 usec
TE 303.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

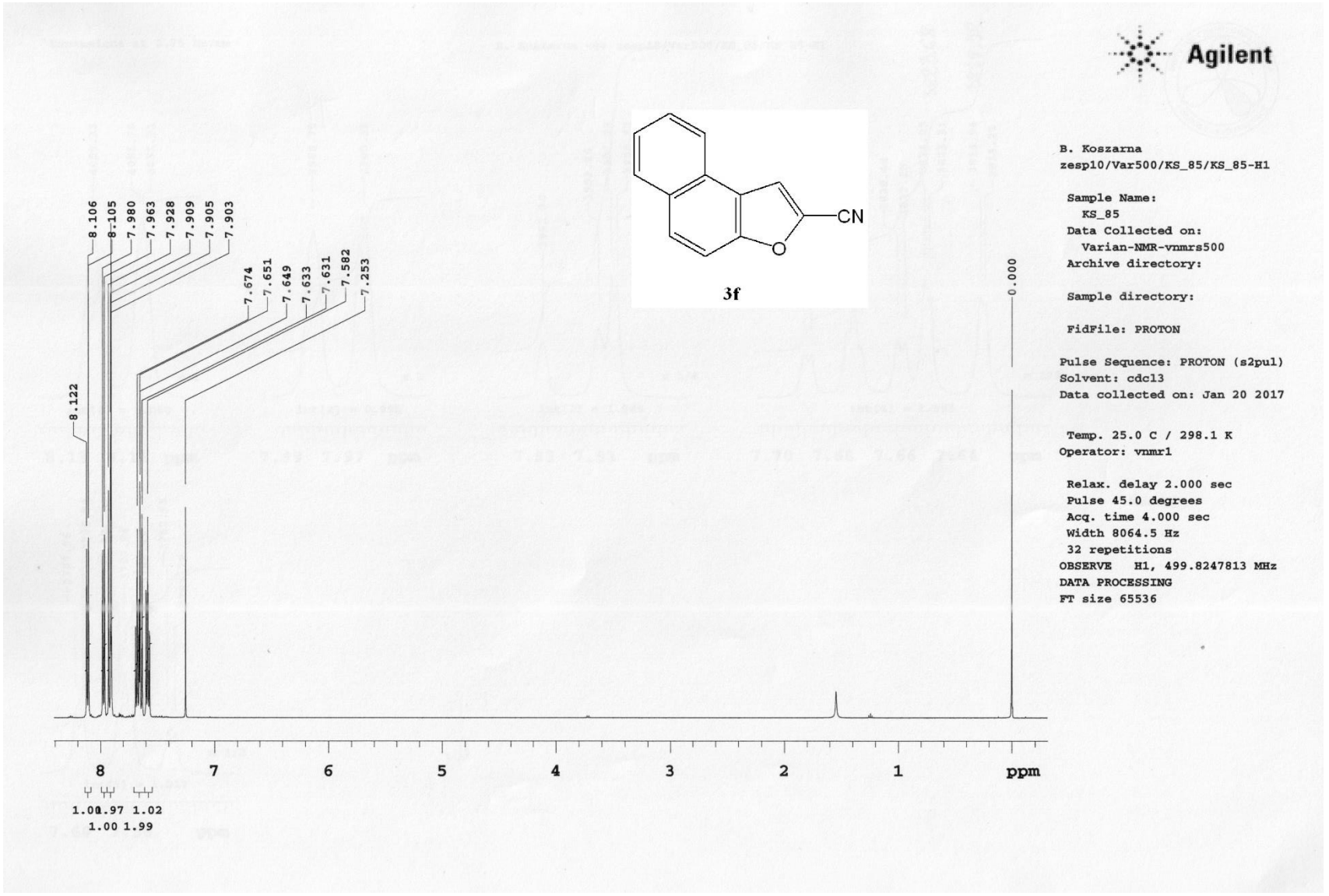
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -3.00 dB
SFO1 125.7703643 MHz

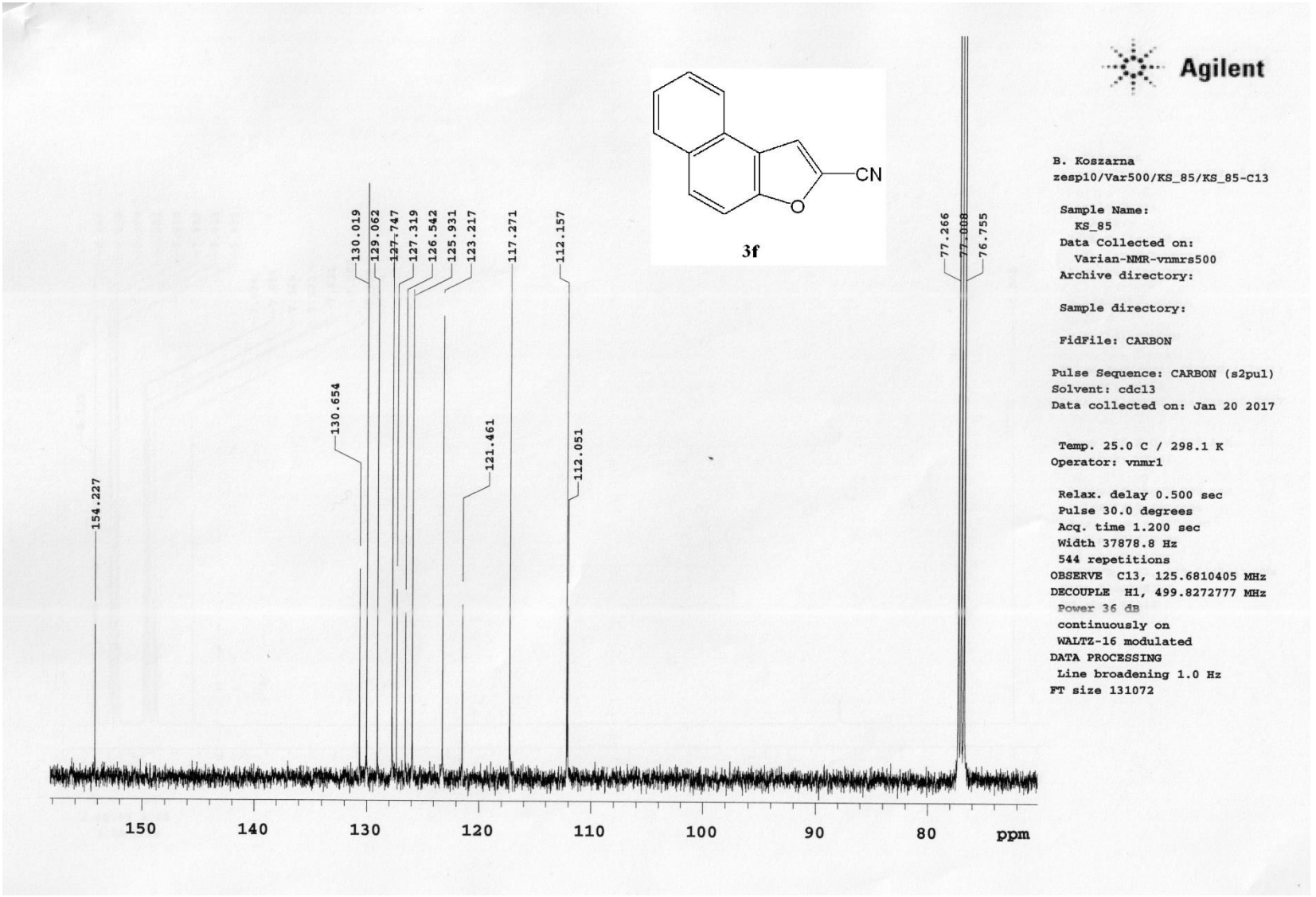
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.00 usec
PL2 3.00 dB
PL12 23.00 dB
PL13 32.00 dB
SFO2 500.1320005 MHz

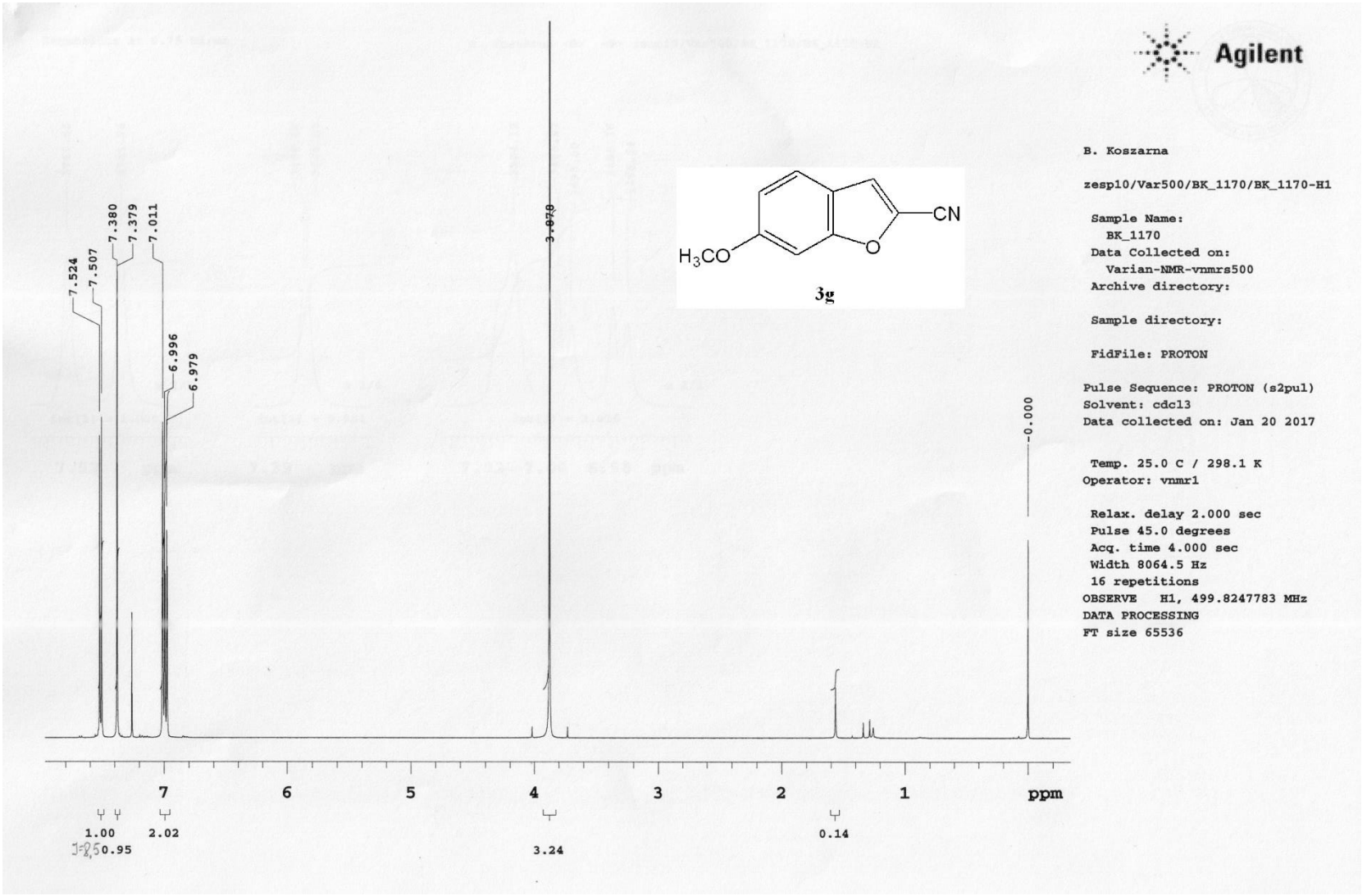
F1 - Acquisition parameters
ND0 1
TD 128
SFO1 500.132 MHz
FIDRES 7.812500 Hz
SW 1.999 ppm
FnMODE QF

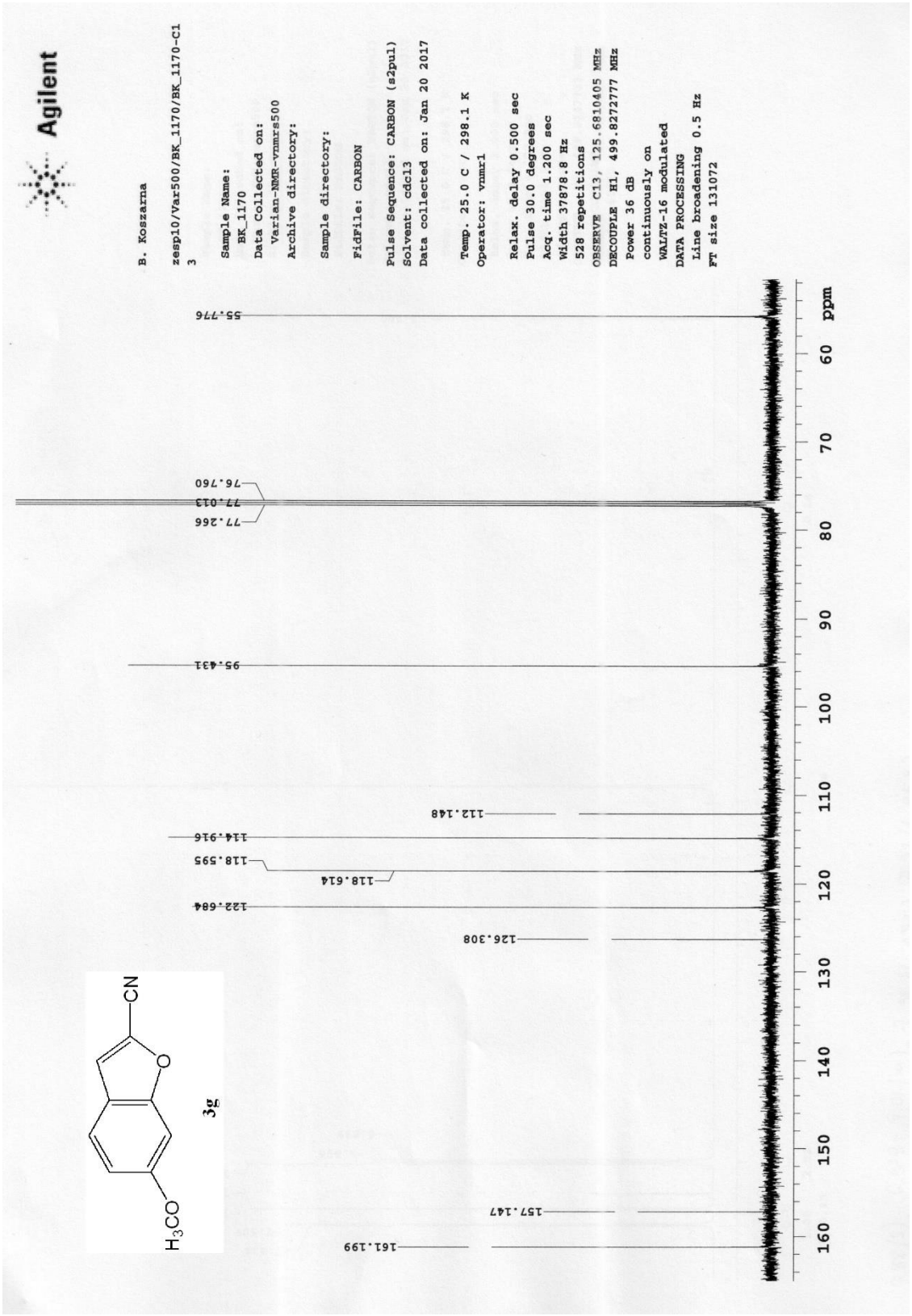
F2 - Processing parameters
SI 262144
SF 125.7577934 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

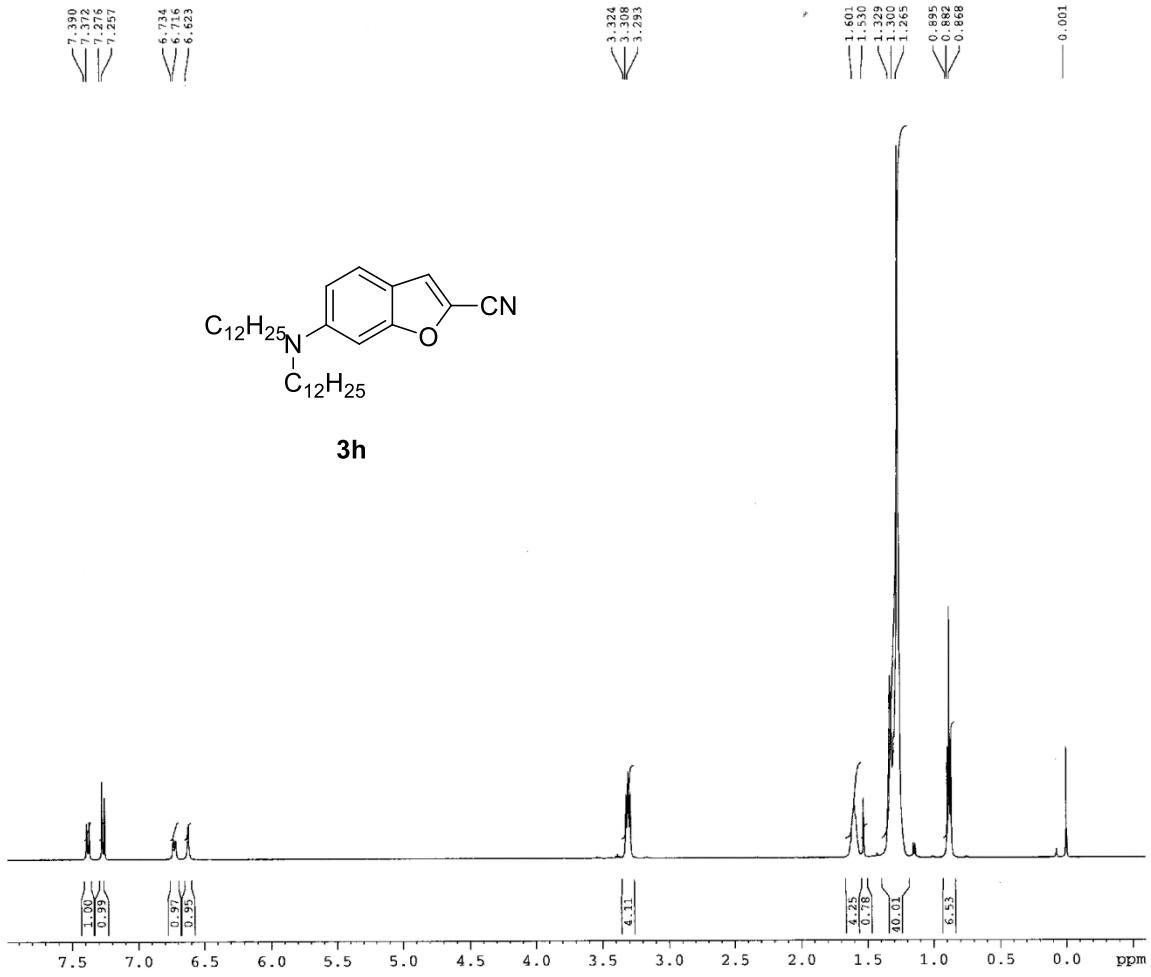
F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0.30 Hz
GB 0.1


Agilent
B. Koszarna
zesp10/Var500/KS_85/KS_85-H1
Sample Name:
KS_85
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 8064.5 Hz
32 repetitions
OBSERVE H1, 499.8247813 MHz
DATA PROCESSING
FT size 65536
CN
O
3f
8.106
8.105
7.980
7.963
7.928
7.909
7.905
7.903
7.674
7.651
7.649
7.633
7.631
7.582
7.253
8.122
0.000
8
7
6
5
4
3
2
1
ppm
1.00
1.97
1.02
1.00
1.99


Agilent
CN
O
3f
154.227
130.654
130.019
129.062
127.747
127.319
126.542
125.931
123.217
121.461
117.271
112.157
112.051
77.266
77.008
76.755
150
140
130
120
110
100
90
80
ppm
B. Koszarna
zesp10/Var500/KS_85/KS_85-C13
Sample Name:
KS_85
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 0.500 sec
Pulse 30.0 degrees
Acq. time 1.200 sec
Width 37878.8 Hz
544 repetitions
OBSERVE C13, 125.6810405 MHz
DECOUPLE H1, 499.8272777 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072


Agilent
B. Koszarna
zesp10/Var500/BK_1170/BK_1170-H1
Sample Name:
BK_1170
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 8064.5 Hz
16 repetitions
OBSERVE H1, 499.8247783 MHz
DATA PROCESSING
FT size 65536
7.524
7.507
7.380
7.379
7.011
6.996
6.979
3.870
0.000
H3CO
CN
O
3g
7
6
5
4
3
2
1
ppm
1.00
0.95
2.02
3.24
0.14


Agilent
B. Koszarna
zesp10/Var500/BK_1170/BK_1170-C13
Sample Name: BK_1170
Data Collected on: Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 0.500 sec
Pulse 30.0 degrees
Acq. time 1.200 sec
Width 37878.8 Hz
528 repetitions
OBSERVE C13, 125.6810405 MHz
DECOUPLE H1, 499.8272777 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
55.776
76.760
77.013
77.266
95.431
112.148
114.916
118.595
118.614
122.684
126.308
157.147
161.199
ppm
CN
O
H3CO
3g



3h

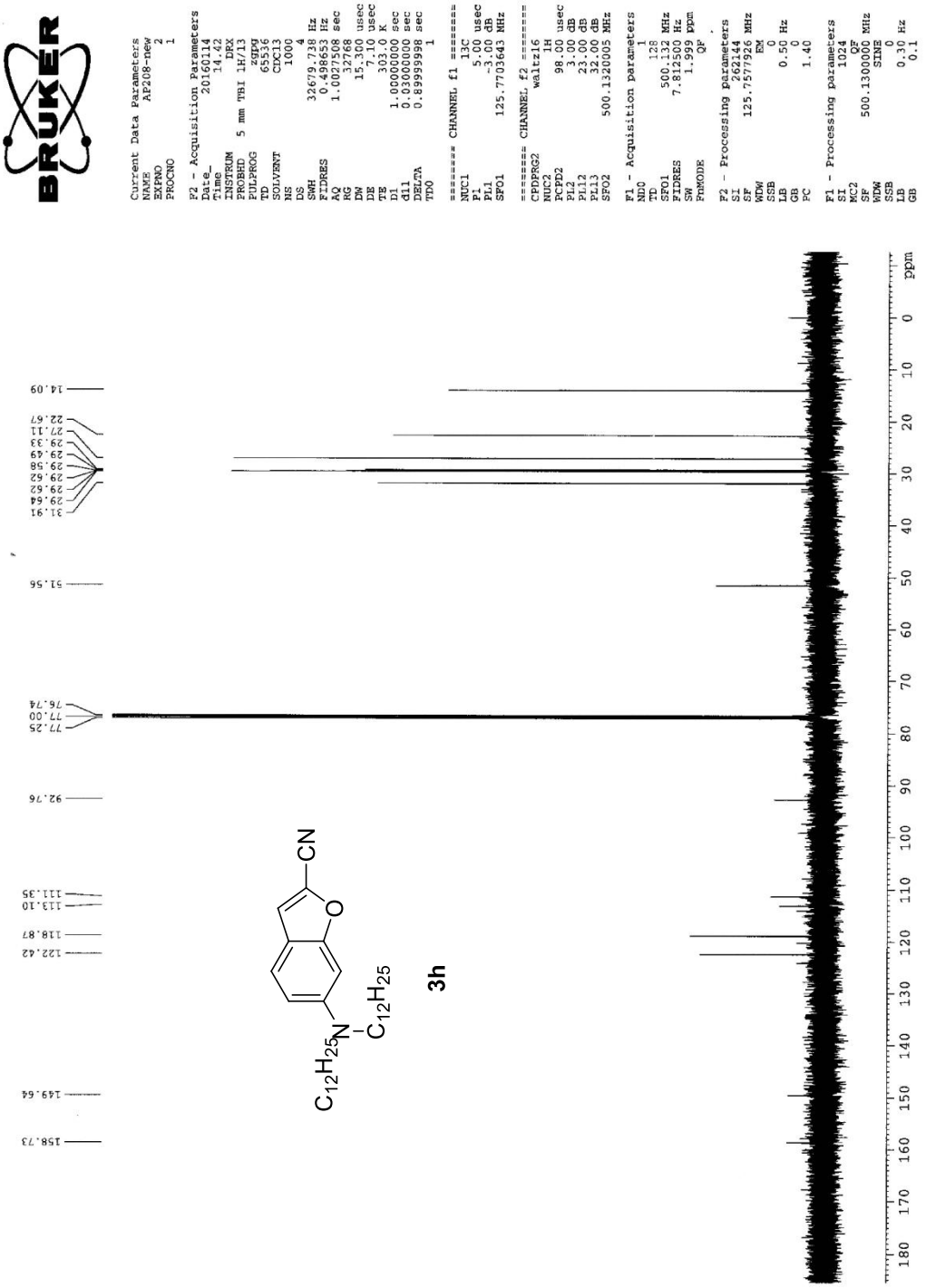
BRUKER

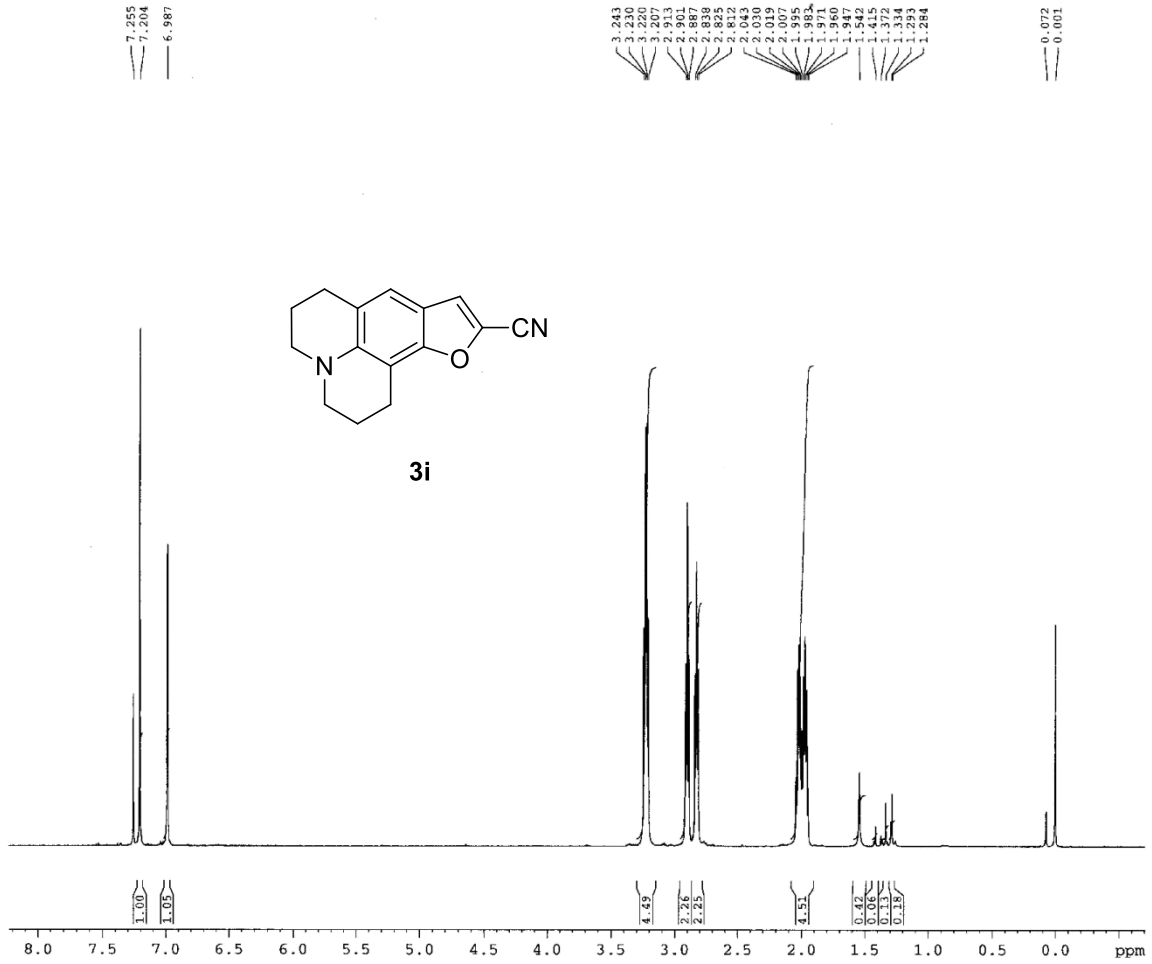
Current Data Parameters
NAME AP208-new
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160114
Time 14.33
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 128
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 500.1330885 MHz

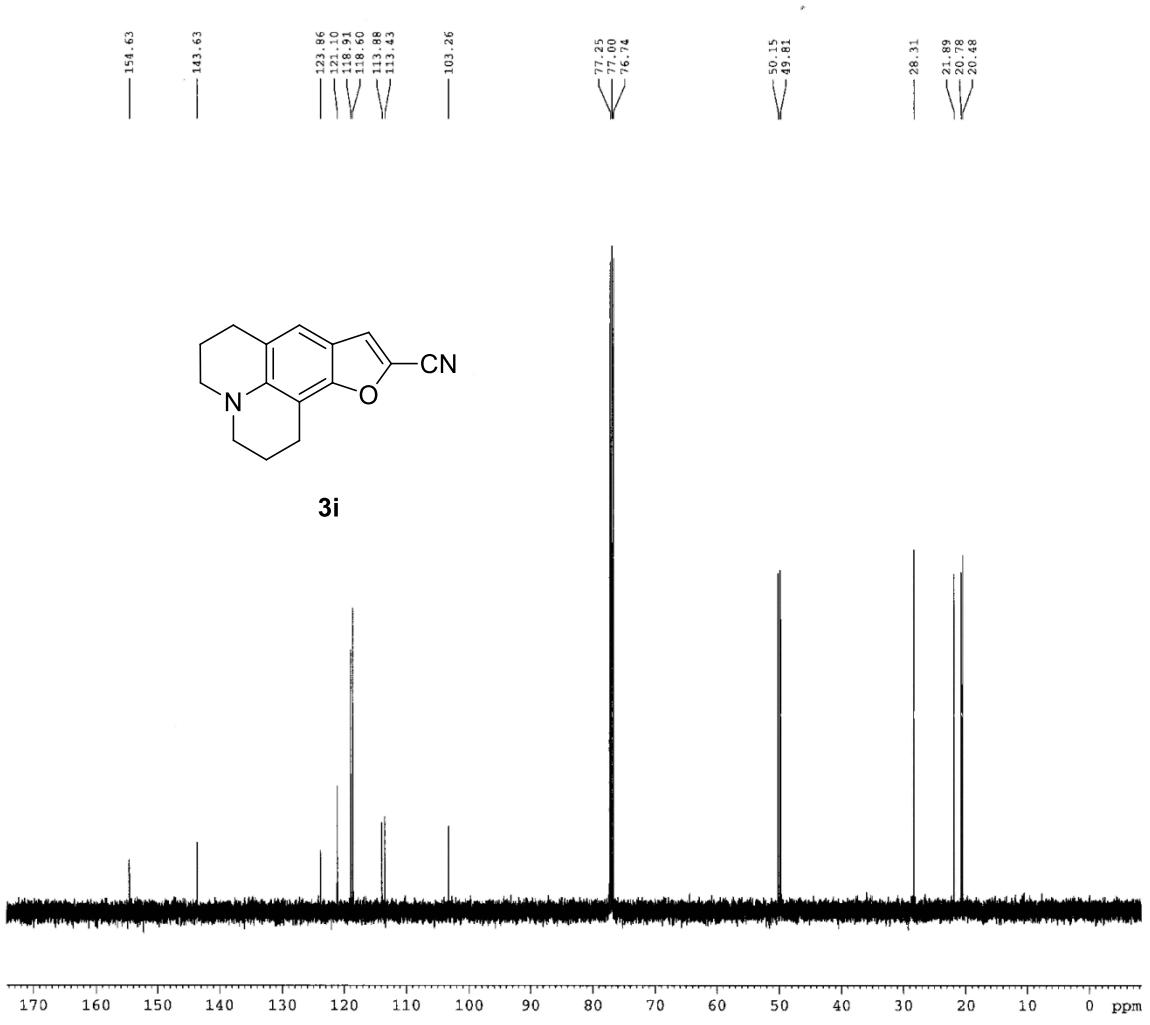
F2 - Processing parameters
SI 32768
SF 500.1300250 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00


Current Data Parameters
NAME AP208-new
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160114
Time 14.42
INSTRUM DRX
PROBHD 5 mm YBI 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1000
DS 4
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027508 sec
RG 32768
DW 15.300 usec
DE 7.10 usec
TE 303.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -3.00 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.00 usec
PL2 3.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 500.1320005 MHz
F1 - Acquisition parameters
ND0 1
TD 128
SFO1 500.132 MHz
FIDRES 7.812500 Hz
SW 1.999 ppm
FnMODE QF
F2 - Processing parameters
SI 262144
SF 125.7577926 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0.30 Hz
GB 0.1
14.09
22.67
27.11
29.33
29.49
29.58
29.62
29.62
29.64
31.91
51.56
76.74
77.00
77.25
92.76
111.35
113.10
118.87
122.42
149.64
158.73
CN
O
N
C12H25
C12H25
3h
ppm



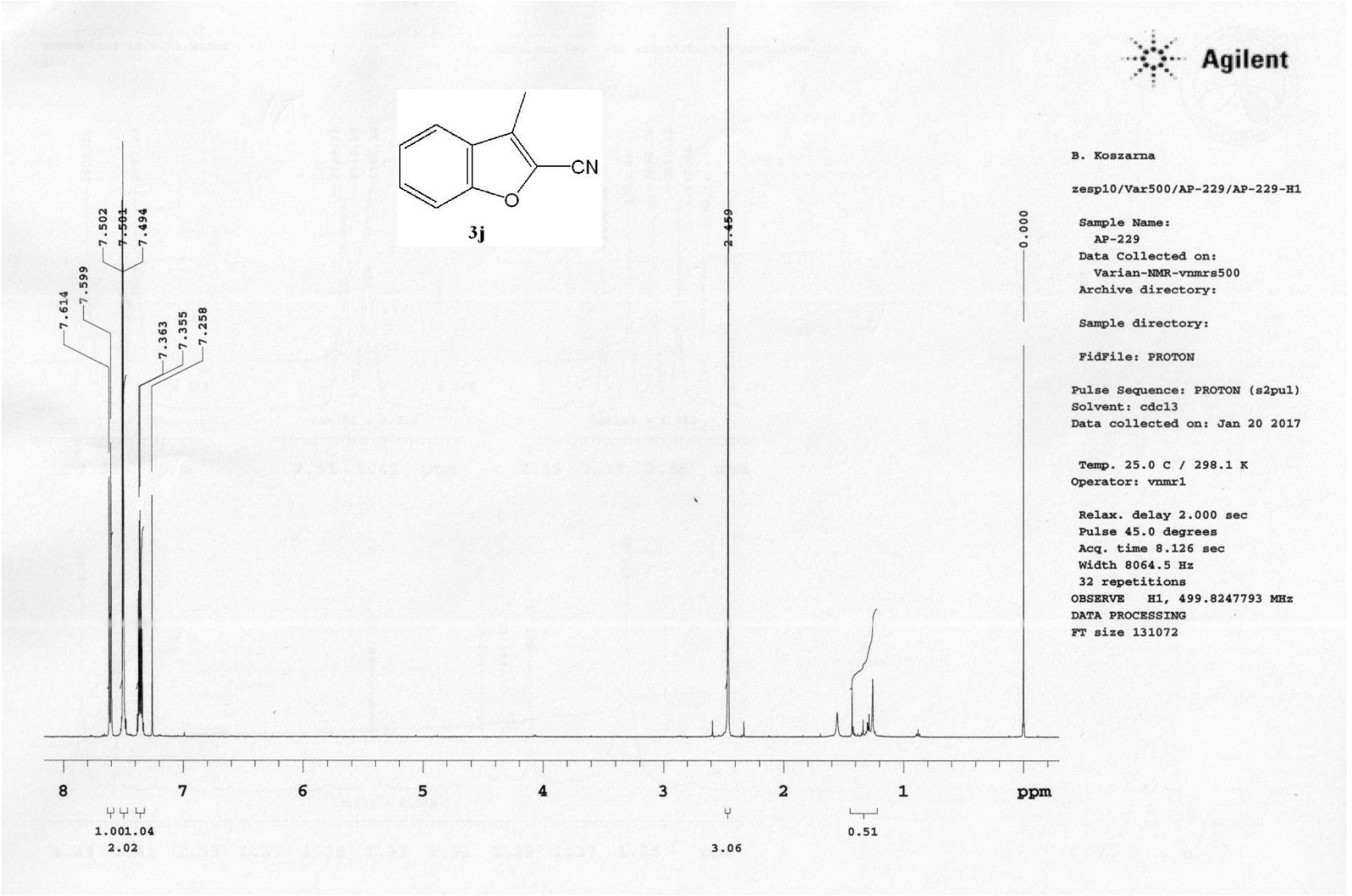
BRUKER

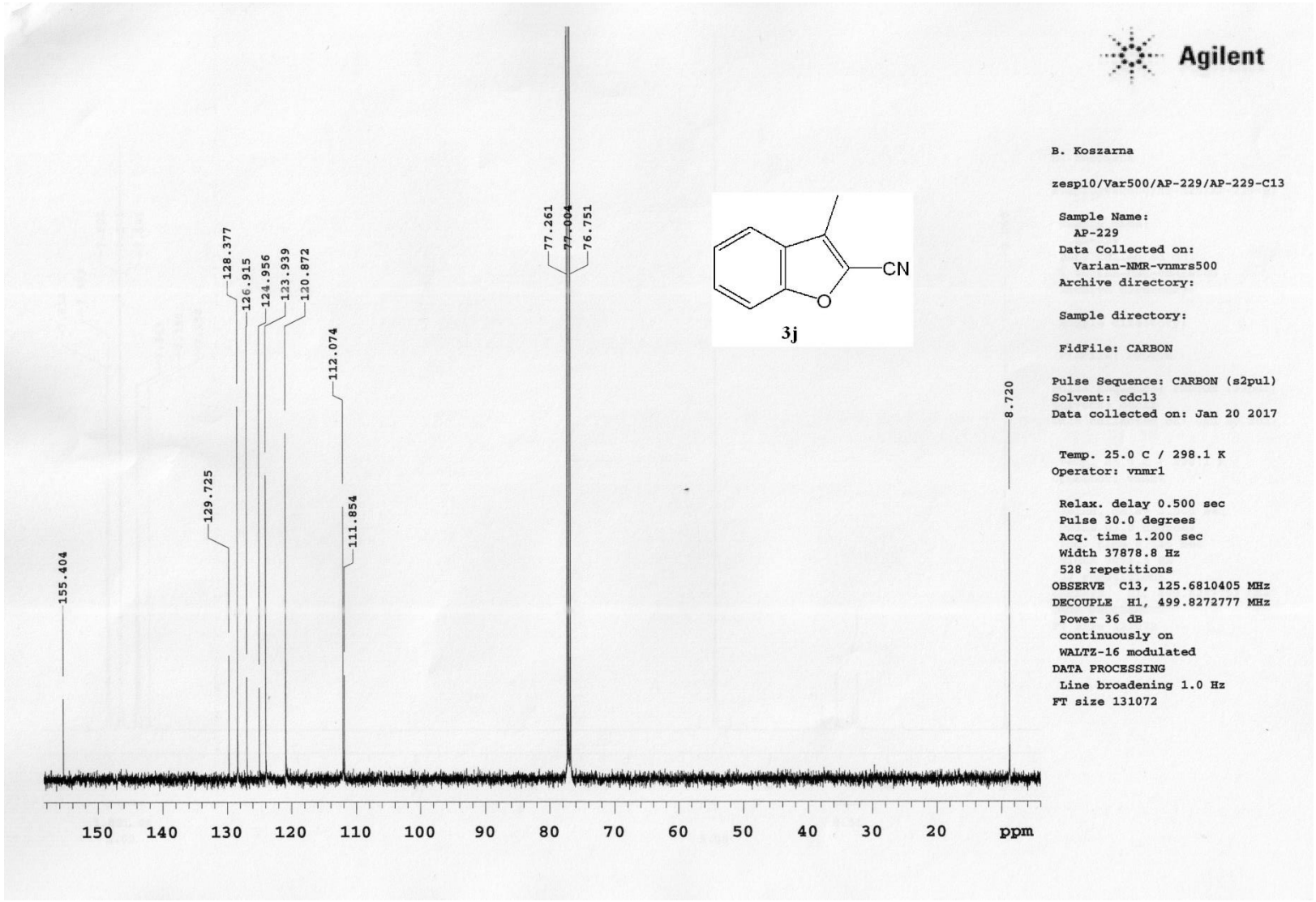
```
Current Data Parameters
NAME                  AP253
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20151202
Time                 11.38
INSTRUM                DRX
PROBHD    5 mm TBI 1H/13
PULPROG                zg
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      0
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1719923 sec
RG                    181
DW               48.400 usec
DE                6.78 usec
TE                303.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  1H
P1                 2.50 usec
PL1                0.00 dB
SFO1        500.1325006 MHz

F2 - Processing parameters
SI                  32768
SF          500.1300263 MHz
WDW                    no
SSB                     0
LB                 0.00 Hz
GB                      0
PC                   1.00
```

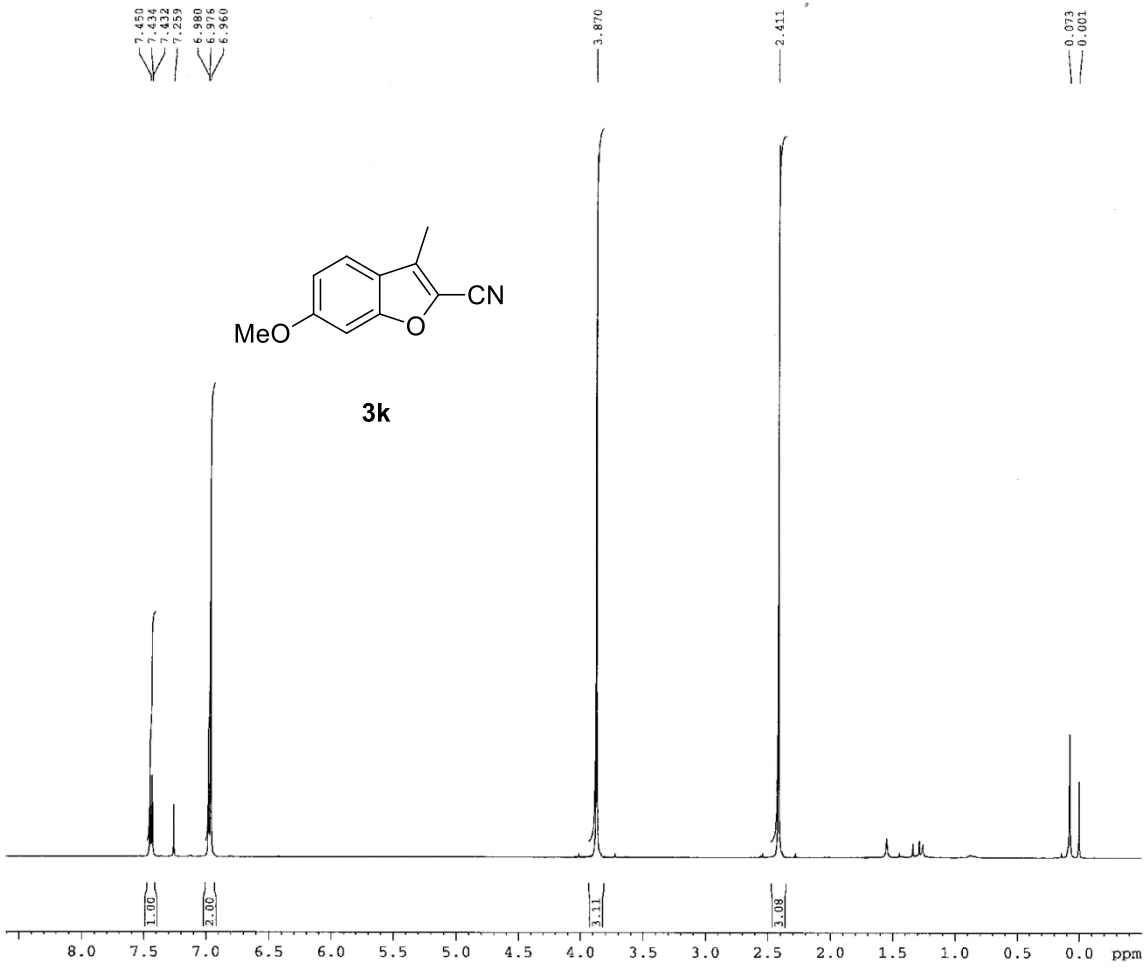


BRUKER

```
Current Data Parameters
NAME                  AP253
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20151202
Time                  11.47
INSTRUM                 DRX
PROBHD    5 mm TBI 1H/13
PULPROG                zgpg
TD                    65536
SOLVENT               CDCl3
NS                     1150
DS                        4
SWH               32679.738 Hz
FIDRES            0.498653 Hz
AQ                1.0027508 sec
RG                    32768
DW                   15.300 usec
DE                    7.10 usec
TE                   303.0 K
D1                1.00000000 sec
d11               0.03000000 sec
DELTA             0.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     5.00 usec
PL1                   -3.00 dB
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 98.00 usec
PL2                    3.00 dB
PL12                  23.00 dB
PL13                  32.00 dB
SFO2           500.1320005 MHz

F1 - Acquisition parameters
ND0                       1
TD                      128
SFO1               500.132 MHz
FIDRES            7.812500 Hz
SW                    1.999 ppm
FnMODE                   QF.

F2 - Processing parameters
SI                    262144
SF             125.7577943 MHz
WDW                     EM
SSB                      0
LB                    0.50 Hz
GB                       0
PC                    1.40

F1 - Processing parameters
SI                      1024
MC2                     QF
SF             500.1300000 MHz
WDW                   SINE
SSB                      0
LB                    0.30 Hz
GB                     0.1
```


Agilent
CN
O
3j
7.614
7.599
7.502
7.501
7.494
7.363
7.355
7.258
2.459
0.000
8
7
6
5
4
3
2
1
ppm
1.00
1.04
2.02
3.06
0.51
B. Koszarna
zesp10/Var500/AP-229/AP-229-H1
Sample Name:
AP-229
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 8.126 sec
Width 8064.5 Hz
32 repetitions
OBSERVE H1, 499.8247793 MHz
DATA PROCESSING
FT size 131072


Agilent
B. Koszarna
zesp10/Var500/AP-229/AP-229-C13
Sample Name:
AP-229
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 20 2017
Temp. 25.0 C / 298.1 K
Operator: vnmr1
Relax. delay 0.500 sec
Pulse 30.0 degrees
Acq. time 1.200 sec
Width 37878.8 Hz
528 repetitions
OBSERVE C13, 125.6810405 MHz
DECOUPLE H1, 499.8272777 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
CN
O
3j
155.404
129.725
128.377
126.915
124.956
123.939
120.872
112.074
111.854
77.261
77.004
76.751
8.720
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm



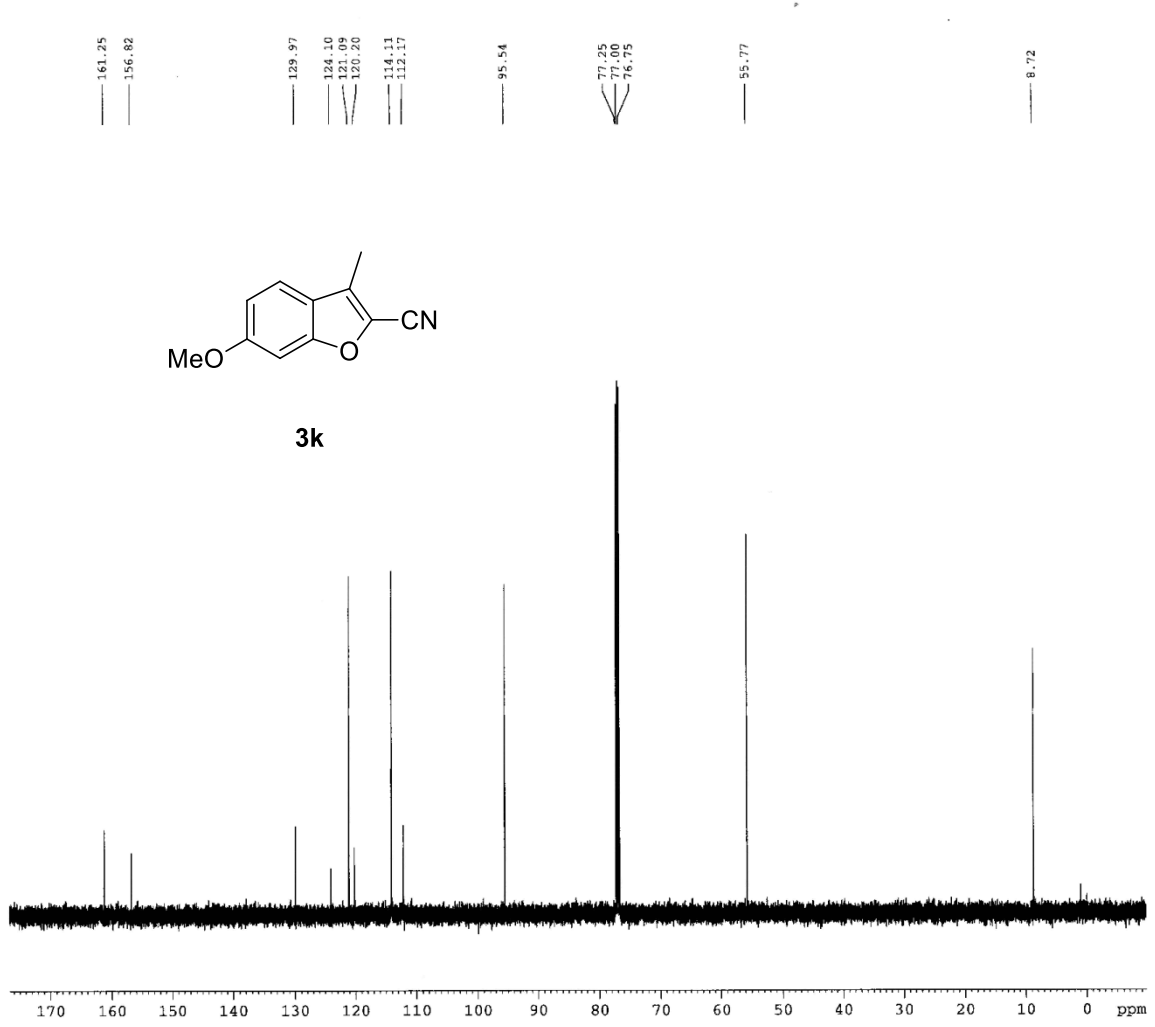
BRUKER

Current Data Parameters
NAME AP245-new
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151126
Time 15.15
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 228.1
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

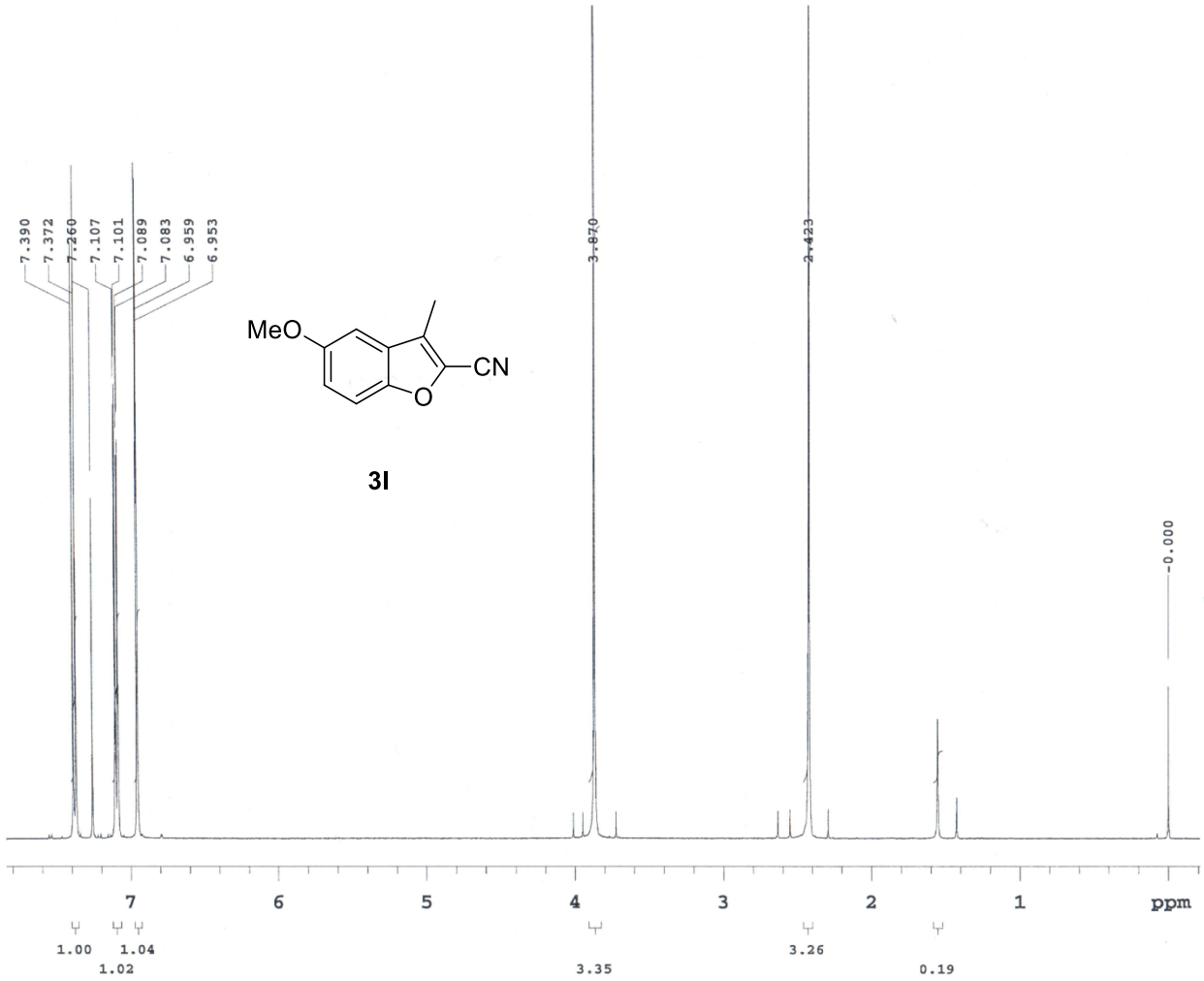
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300238 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



BRUKER

```
Current Data Parameters
NAME                  AP245-new
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20151126
Time                      15.46
INSTRUM                     DRX
PROBHD    5 mm TBI 1H/13
PULPROG                    zgpg
TD                        65536
SOLVENT                   CDCl3
NS                          880
DS                            4
SWH                   32679.738 Hz
FIDRES                0.498653 Hz
AQ                    1.0027508 sec
RG                        32768
DW                       15.300 usec
DE                        7.10 usec
TE                       303.0 K
D1                    1.00000000 sec
d11                   0.03000000 sec
DELTA                 0.89999998 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                         5.00 usec
PL1                       -3.00 dB
SFO1                125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     98.00 usec
PL2                        3.00 dB
PL12                      23.00 dB
PL13                      32.00 dB
SFO2                500.1320005 MHz

F1 - Acquisition parameters
ND0                           1
TD                          128
SFO1                  500.132 MHz
FIDRES                7.812500 Hz
SW                       1.999 ppm
FnMODE                       QF

F2 - Processing parameters
SI                       262144
SF                  125.7577935 MHz
WDW                         EM
SSB                          0
LB                        0.50 Hz
GB                           0
PC                        1.40

F1 - Processing parameters
SI                         1024
MC2                          QF
SF                  500.1300000 MHz
WDW                       SINE
SSB                          0
LB                        0.30 Hz
GB                         0.1
```



3l

Agilent

A. Purc

zesp10/Var500/AP260/AP260-H1

Sample Name:
AP260
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:

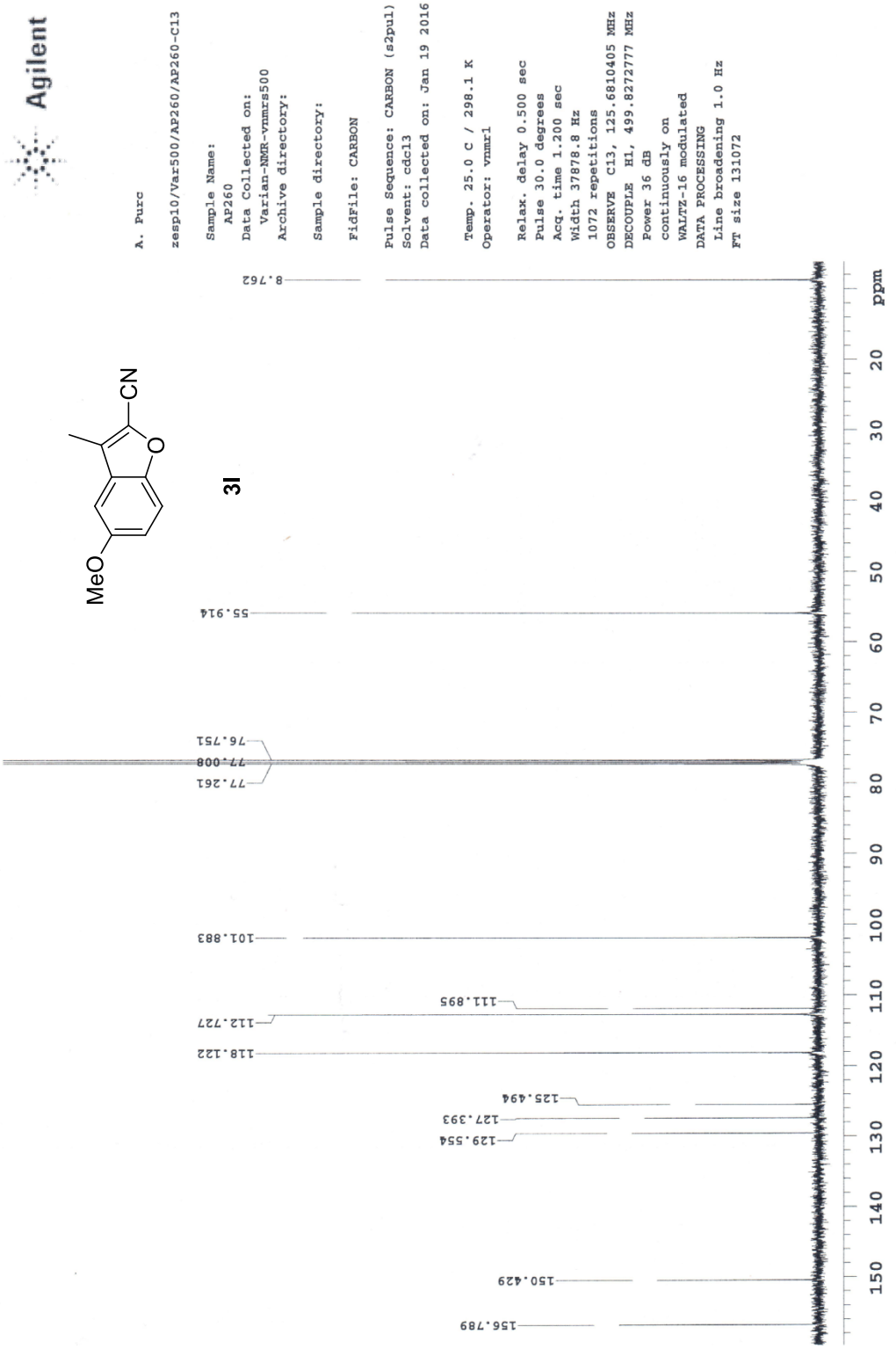
Sample directory:

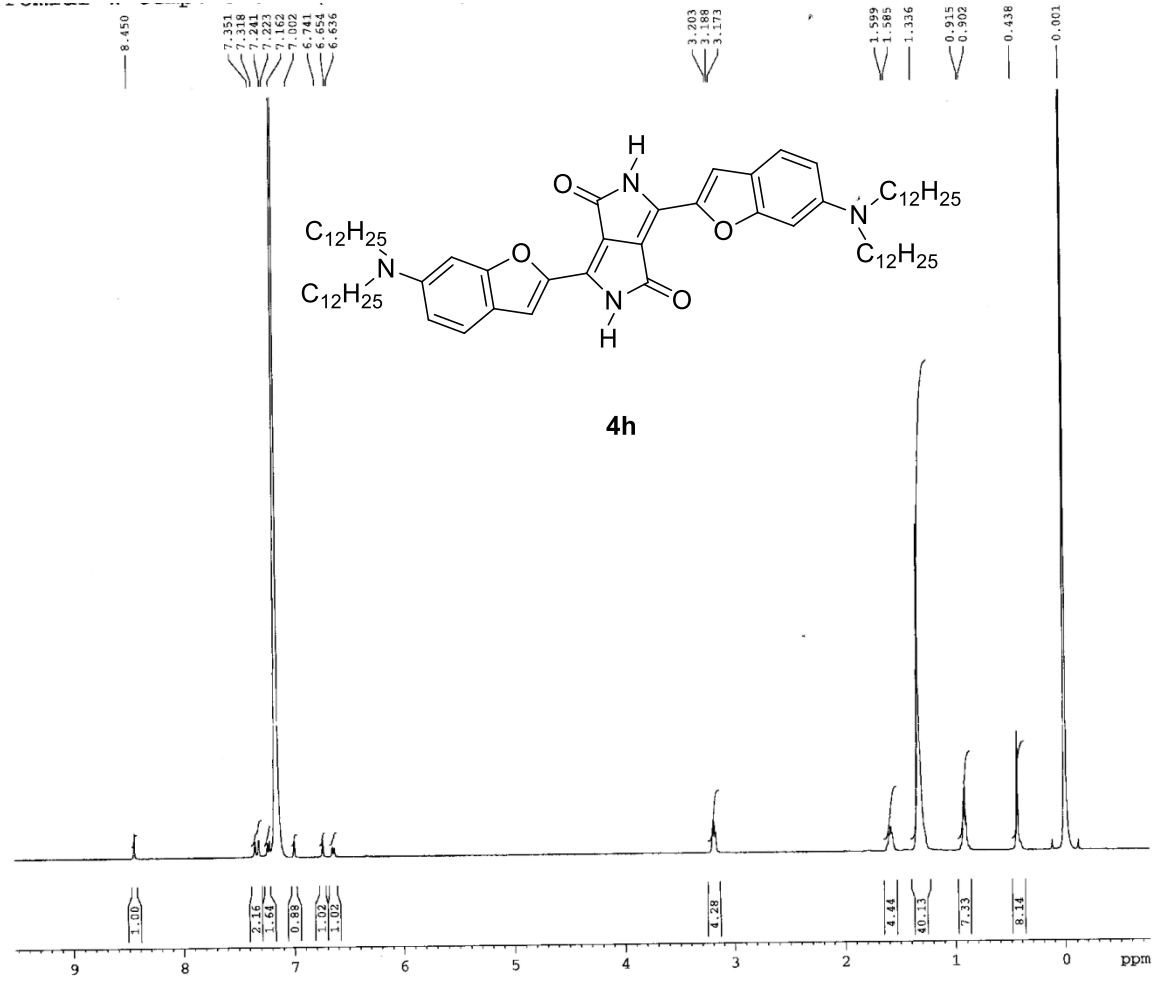
FidFile: PROTON

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 19 2016

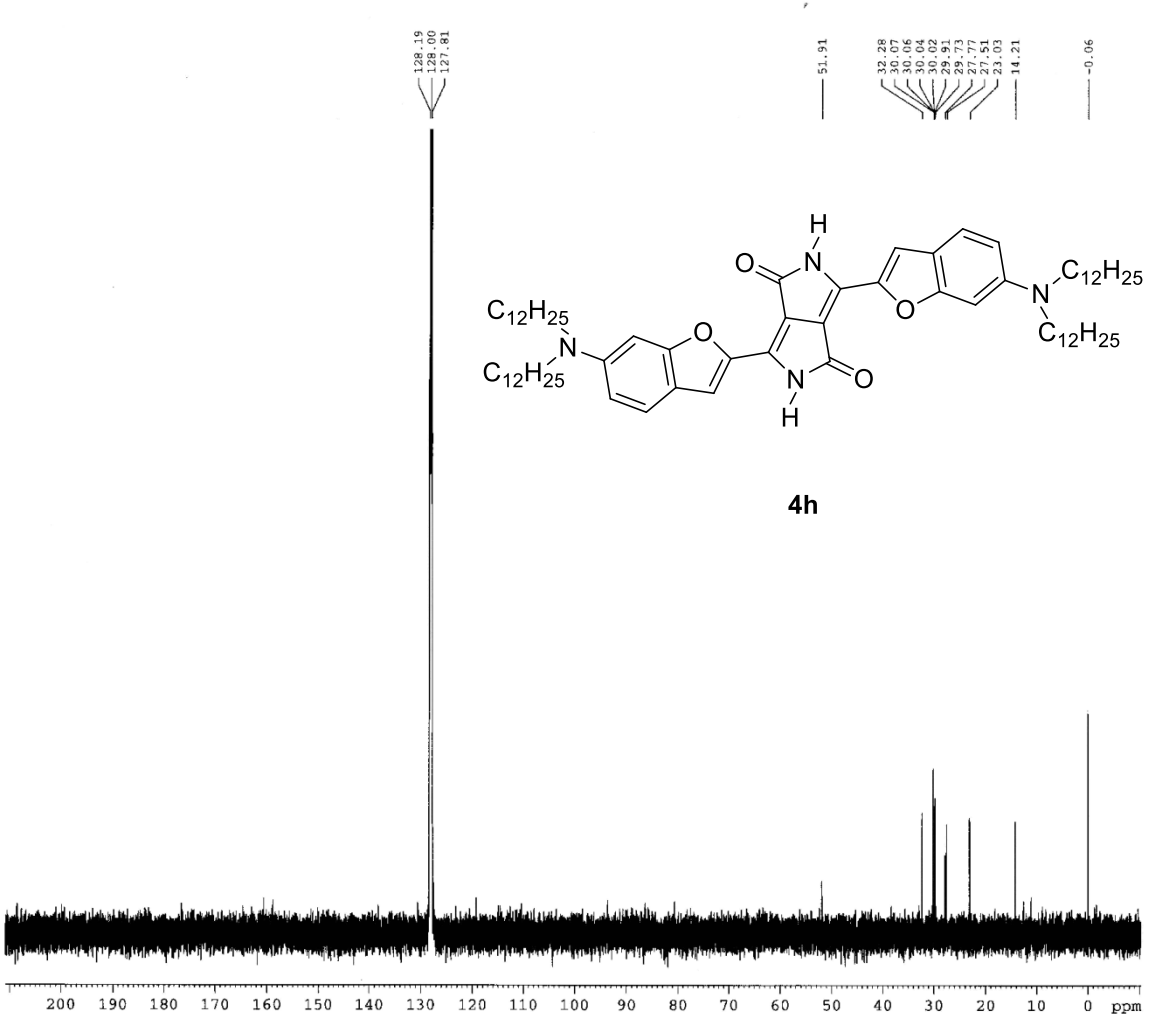
Temp. 25.0 C / 298.1 K
Operator: vnmr1

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.303 sec
Width 9920.6 Hz
20 repetitions
OBSERVE H1, 499.8247780 MHz
DATA PROCESSING
FT size 65536


Agilent
A. Purc
zesp10/Var500/AP260/AP260-C13
Sample Name:
AP260
Data Collected on:
Varian-NMR-vnmrs500
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 19 2016
Temp. 25.0 C / 298.1 K
Operator: vnmrl
Relax. delay 0.500 sec
Pulse 30.0 degrees
Acq. time 1.200 sec
Width 37878.8 Hz
1072 repetitions
OBSERVE C13, 125.6810405 MHz
DECOUPLE H1, 499.8272777 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
CN
O
MeO
3l
8.762
55.914
76.751
77.008
77.261
101.883
111.895
112.727
118.122
125.494
127.393
129.554
150.429
156.789
ppm



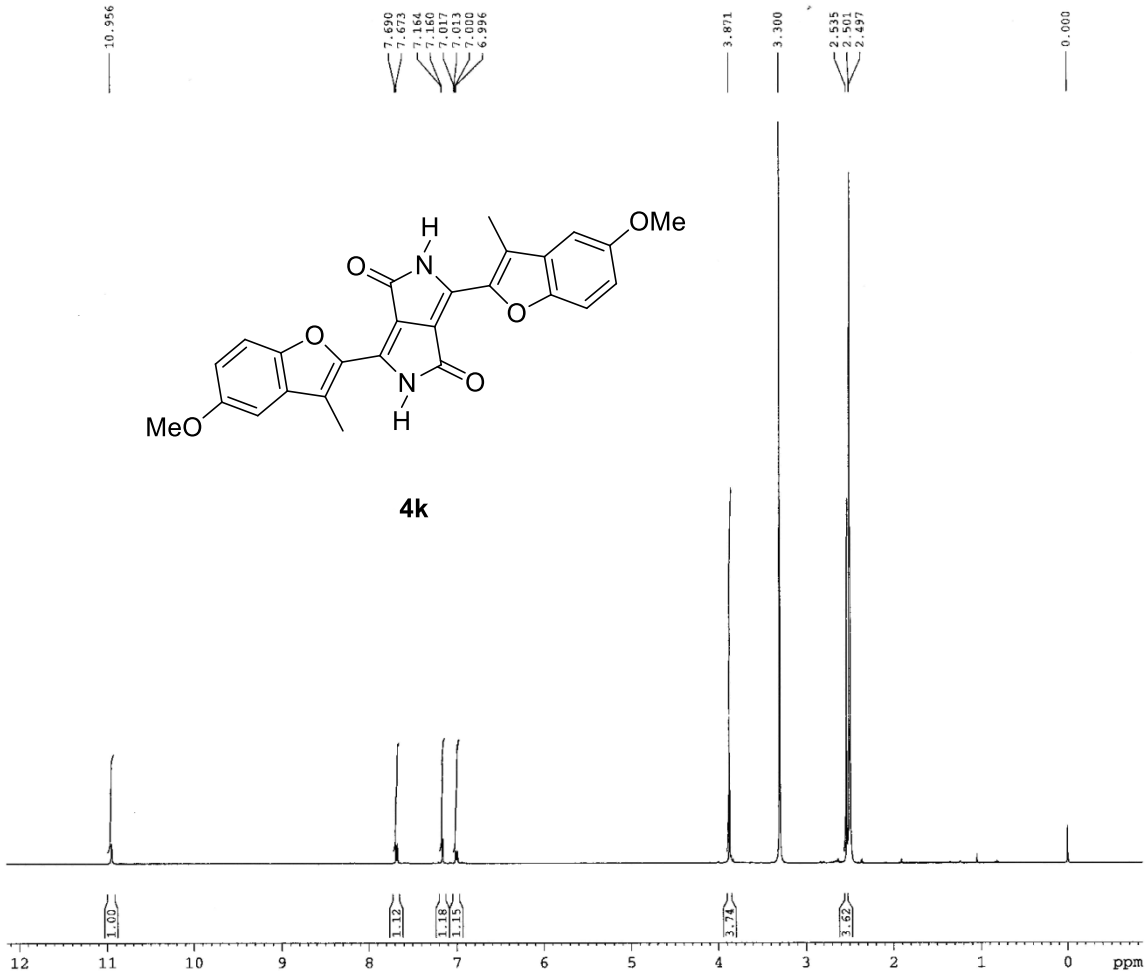
```
Current Data Parameters
NAME                  AP213-C6H6
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20160211
Time                       12.40
INSTRUM                      DRX
PROBHD    5 mm TBI 1H/13
PULPROG                     zg30
TD                         65536
SOLVENT                    C6D6
NS                          128
DS                             0
SWH                    10330.578 Hz
FIDRES                 0.157632 Hz
AQ                     3.1719923 sec
RG                         645.1
DW                        48.400 usec
DE                         6.78 usec
TE                        343.0 K
D1                     1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                         8.20 usec
PL1                        5.00 dB
SFO1                500.1330885 MHz

F2 - Processing parameters
SI                         32768
SF                  500.1300549 MHz
WDW                           EM
SSB                            0
LB                          0.50 Hz
GB                             0
PC                          1.00
```



4h

BRUKER

```
Current Data Parameters
NAME                  AP213-temp
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20160301
Time                       11.13
INSTRUM                      DRX
PROBHD    5 mm TBI 1H/13
PULPROG                   zgpg
TD                           65536
SOLVENT                      C6D6
NS                           7000
DS                              4
SWH                  32679.738 Hz
FIDRES                0.498653 Hz
AQ                    1.0027508 sec
RG                           32768
DW                       15.300 usec
DE                        7.10 usec
TE                       323.0 K
D1                    1.00000000 sec
d11                   0.03000000 sec
DELTA                 0.89999998 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                         13C
P1                        5.00 usec
PL1                      -3.00 dB
SFO1                125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                    98.00 usec
PL2                       3.00 dB
PL12                     23.00 dB
PL13                     32.00 dB
SFO2                500.1320005 MHz

F1 - Acquisition parameters
ND0                             1
TD                            128
SFO1                  500.132 MHz
FIDRES                7.812500 Hz
SW                       1.999 ppm
FnMODE                      QF

F2 - Processing parameters
SI                         262144
SF                  125.7577560 MHz
WDW                          EM
SSB                           0
LB                         0.50 Hz
GB                            0
PC                         1.40

F1 - Processing parameters
SI                          1024
MC2                           QF
SF                  500.1300000 MHz
WDW                        SINE
SSB                           0
LB                         0.30 Hz
GB                          0.1
```



BRUKER

Current Data Parameters

NAME	AP248
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

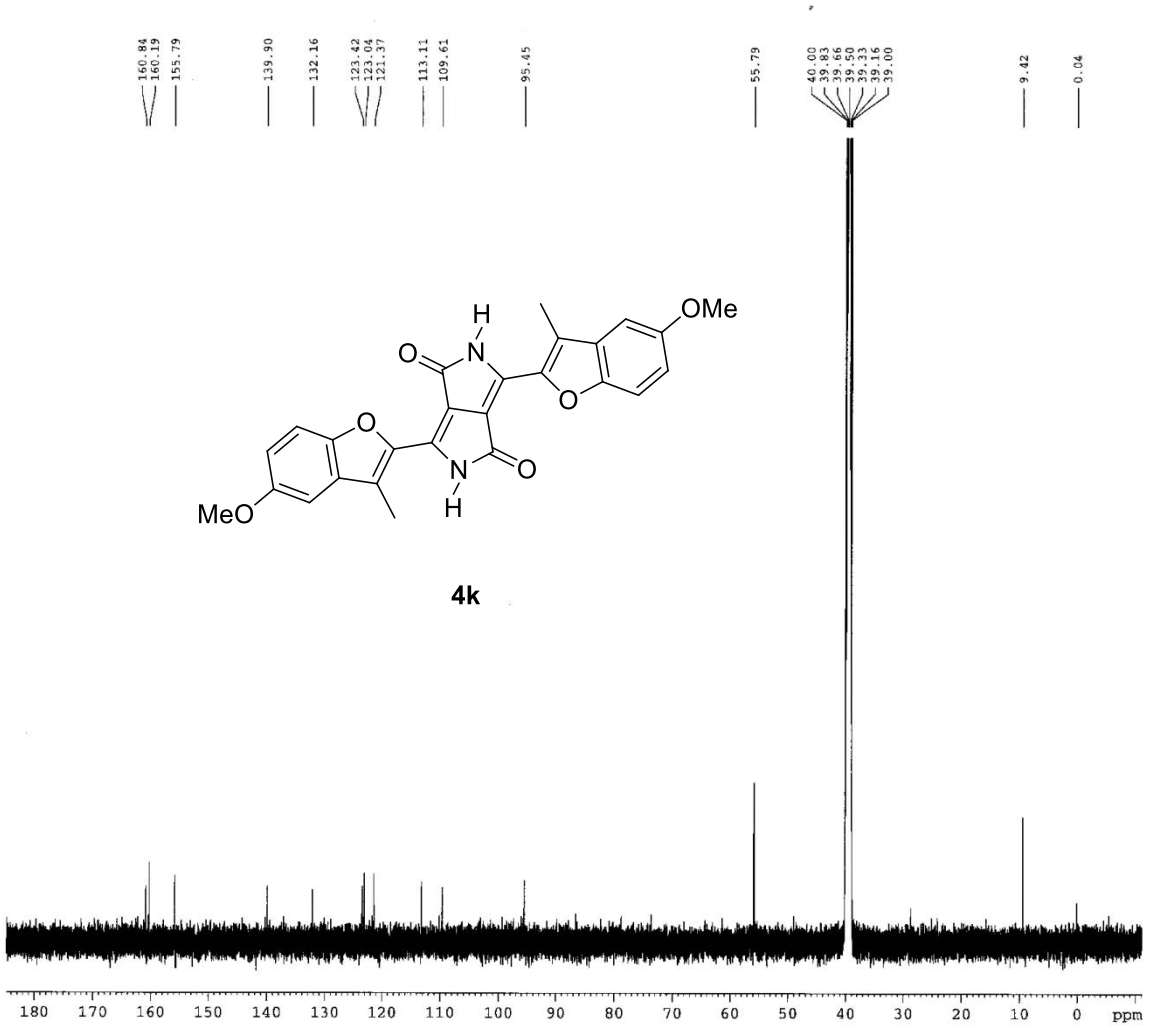
Date_	20151126
Time	17.12
INSTRUM	DRX
PROBHD	5 mm TBI 1H/13
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	32
DS	0
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	406.4
DW	48.400 usec
DE	6.78 usec
TE	303.0 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	8.20 usec
PL1	5.00 dB
SFO1	500.1330885 MHz

F2 - Processing parameters

SI	32768
SF	500.1300042 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



4k

BRUKER

Current Data Parameters
NAME AP248
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151126
Time 17.46
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 30000
DS 4
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027508 sec
RG 101.6
DW 15.300 usec
DE 7.10 usec
TE 303.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

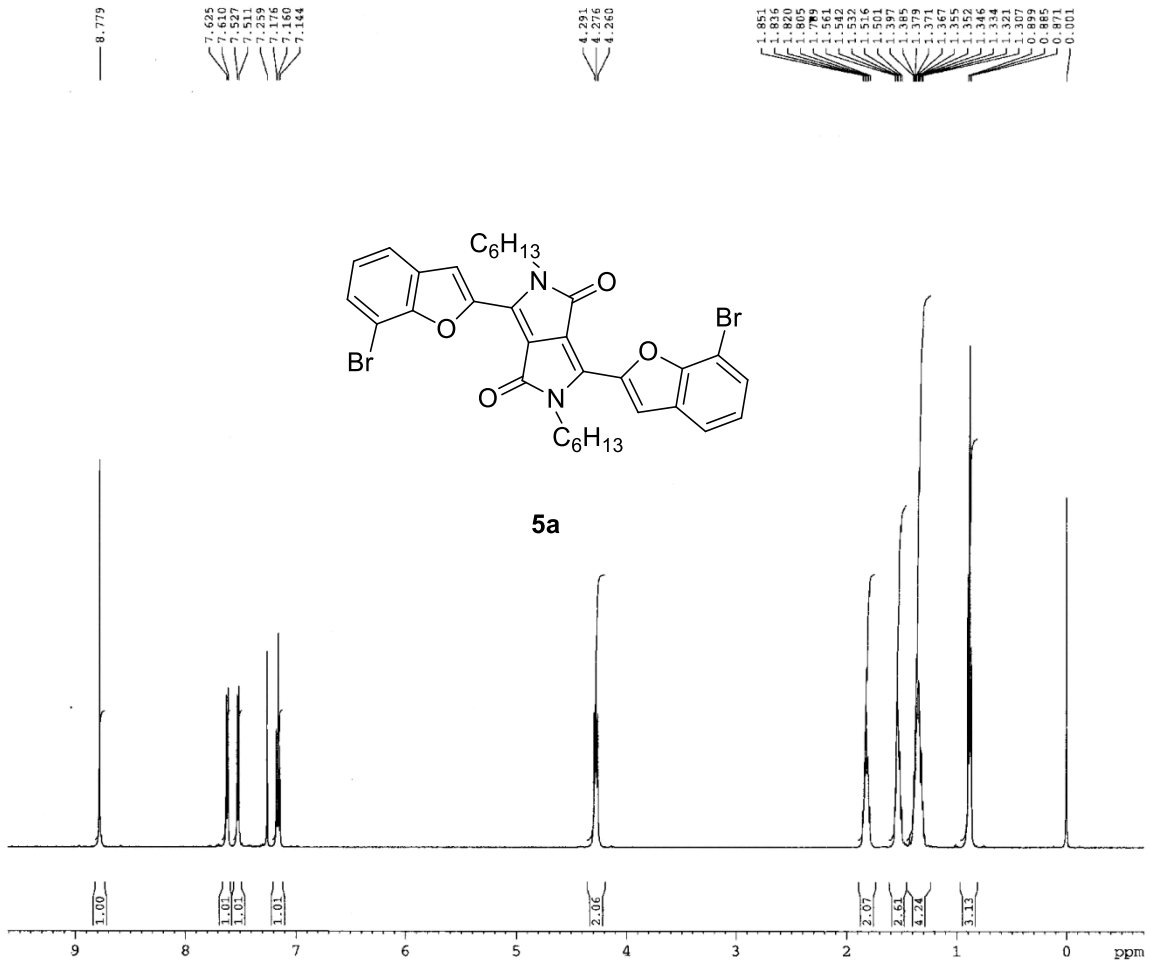
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -3.00 dB
SFO1 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.00 usec
PL2 3.00 dB
PL12 23.00 dB
PL13 32.00 dB
SFO2 500.1320005 MHz

F1 - Acquisition parameters
ND0 1
TD 128
SFO1 500.132 MHz
FIDRES 7.812500 Hz
SW 1.999 ppm
FnMODE QF

F2 - Processing parameters
SI 262144
SF 125.7578568 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0.30 Hz
GB 0.1



5a

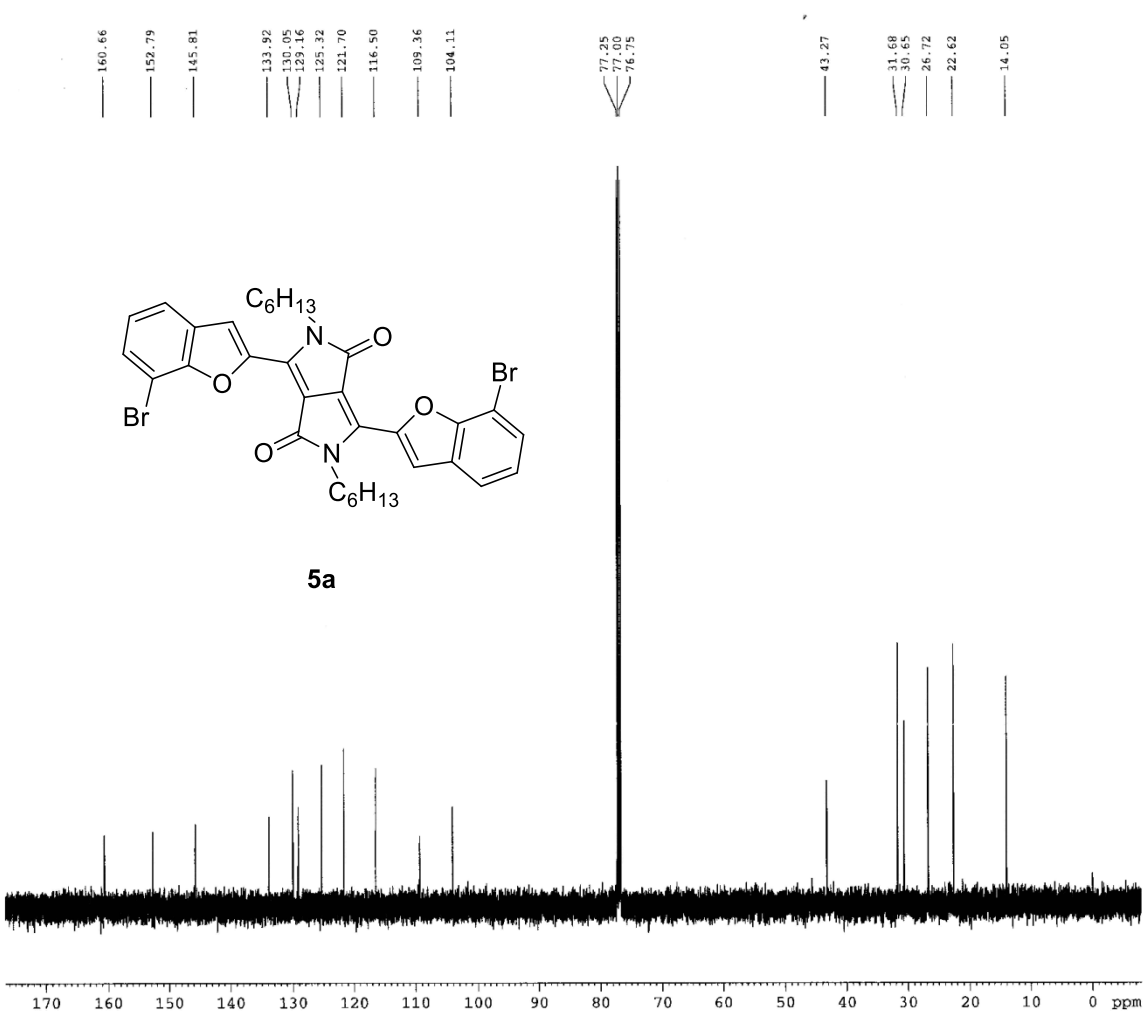
BRUKER

Current Data Parameters
NAME AP256
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160114
Time 16.28
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 287.4
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 500.1330885 MHz

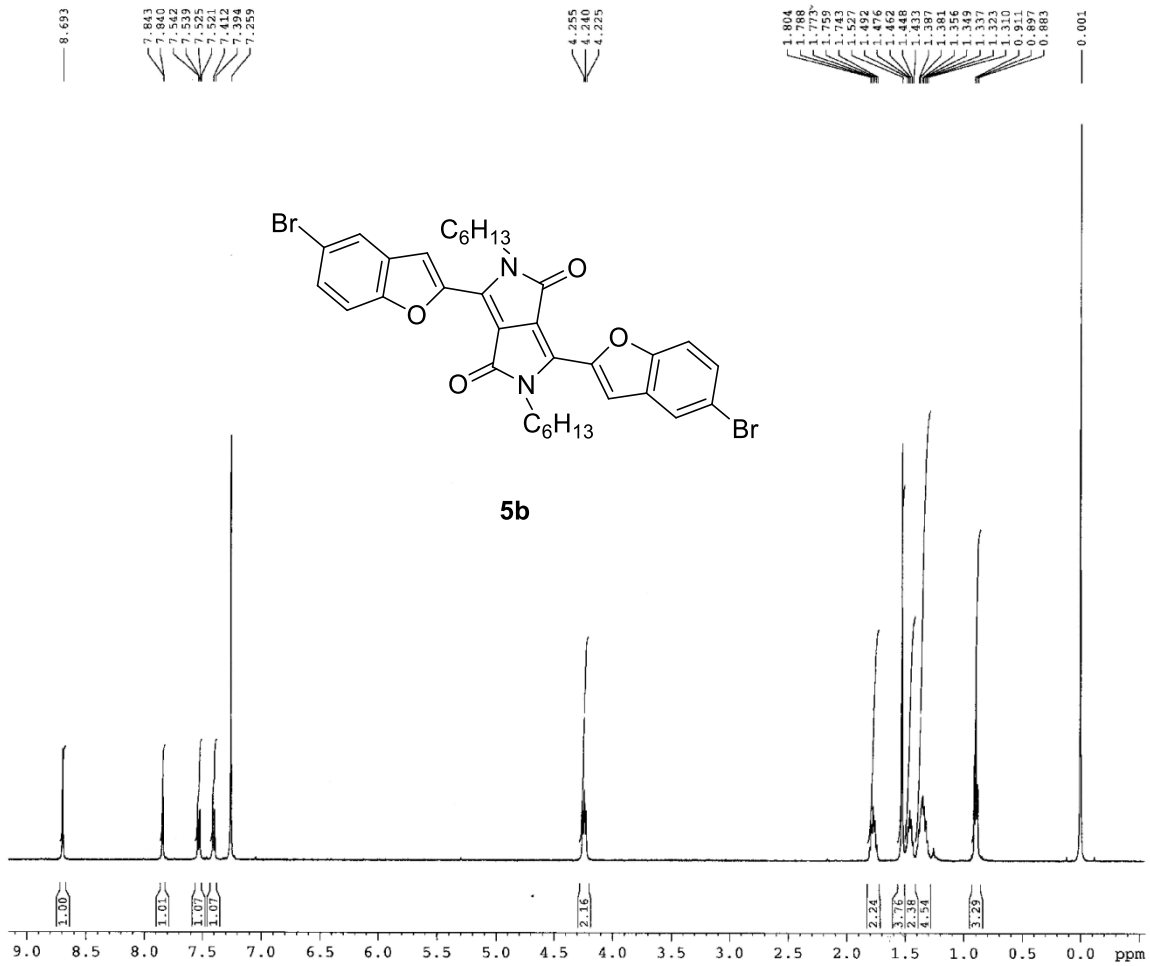
F2 - Processing parameters
SI 32768
SF 500.1300240 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



5a

BRUKER

```
Current Data Parameters
NAME                  AP256
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20160114
Time                  16.57
INSTRUM                 DRX
PROBHD   5 mm TBI 1H/13
PULPROG                zgpg
TD                    65536
SOLVENT               CDCl3
NS                      760
DS                        4
SWH               32679.738 Hz
FIDRES            0.498653 Hz
AQ                1.0027508 sec
RG                    32768
DW                   15.300 usec
DE                    7.10 usec
TE                   303.0 K
D1                1.00000000 sec
d11               0.03000000 sec
DELTA             0.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     5.00 usec
PL1                   -3.00 dB
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 98.00 usec
PL2                    3.00 dB
PL12                  23.00 dB
PL13                  32.00 dB
SFO2           500.1320005 MHz

F1 - Acquisition parameters
ND0                       1
TD                      128
SFO1              500.132 MHz
FIDRES            7.812500 Hz
SW                    1.999 ppm
FnMODE                   QF

F2 - Processing parameters
SI                   262144
SF             125.7577927 MHz
WDW                      EM
SSB                       0
LB                     0.50 Hz
GB                        0
PC                     1.40

F1 - Processing parameters
SI                     1024
MC2                      QF
SF             500.1300000 MHz
WDW                    SINE
SSB                       0
LB                     0.30 Hz
GB                      0.1
```



5b

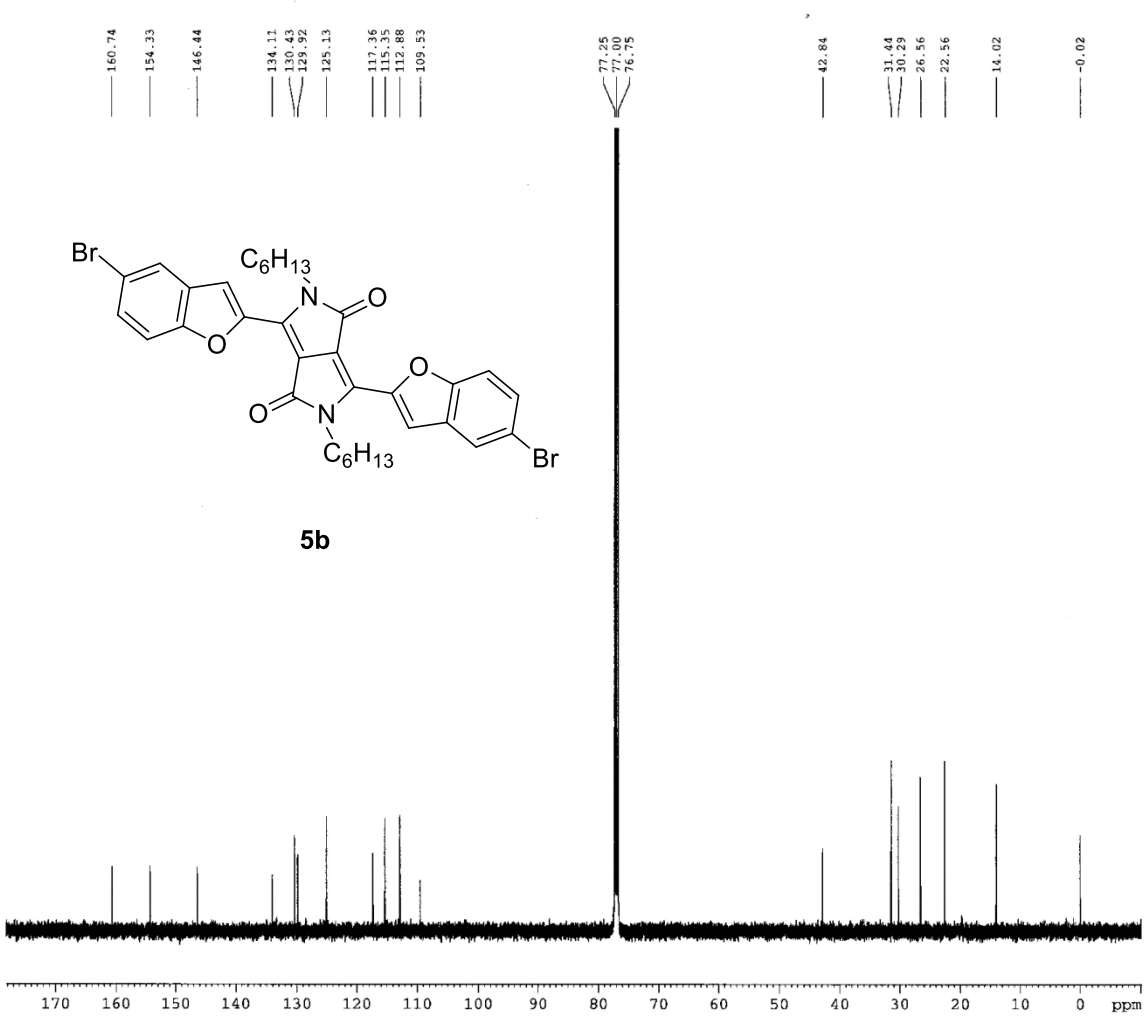
BRUKER

Current Data Parameters
NAME BK-1161-new
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160118
Time 10.26
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 574.7
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

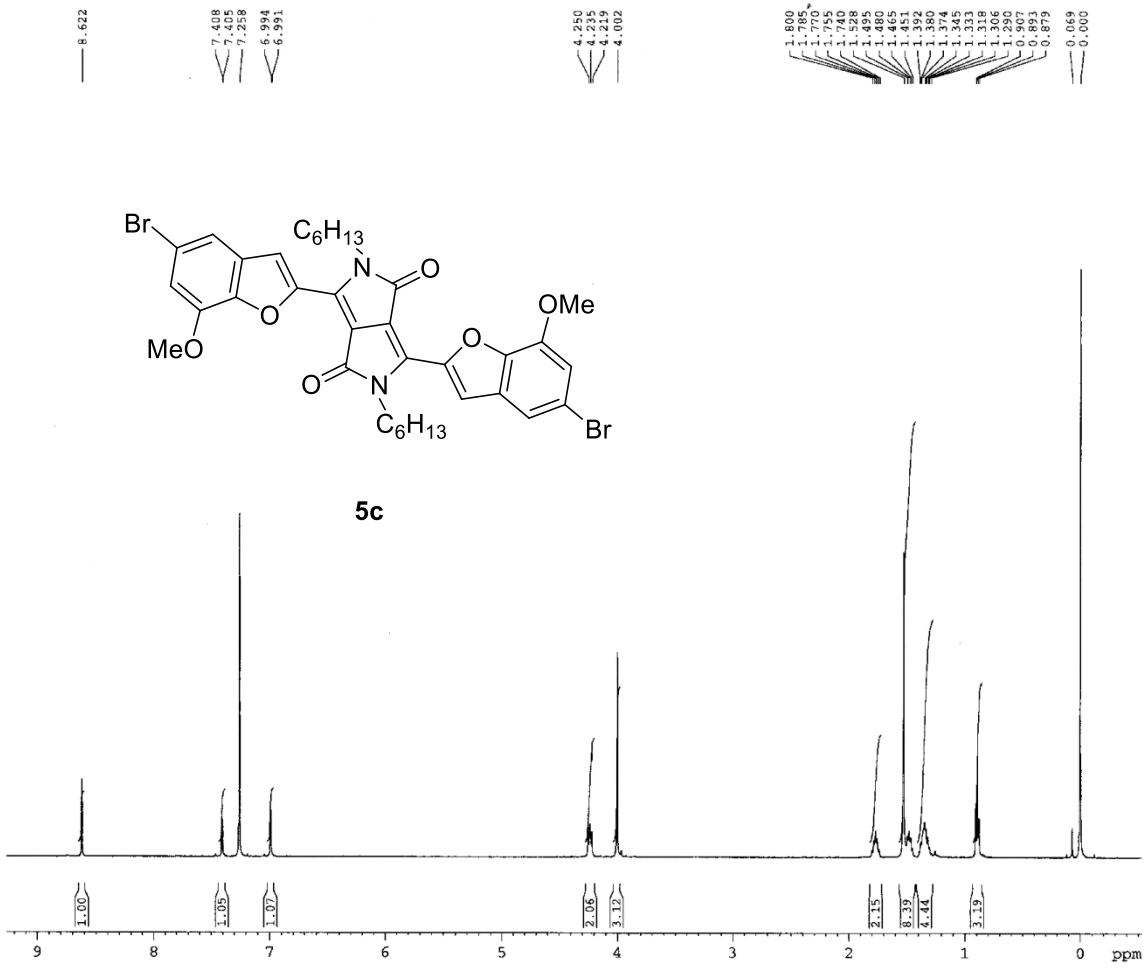
======== CHANNEL f1 ========
NUC1 1H
P1 2.50 usec
PL1 0.00 dB
SFO1 500.1325006 MHz

F2 - Processing parameters
SI 32768
SF 500.1300244 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



5b

```
Current Data Parameters
NAME                  BK-1161-new
EXPNO                           2
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20160119
Time                        14.12
INSTRUM                       DRX
PROBHD    5 mm TBI 1H/13
PULPROG                     zgpg
TD                         65536
SOLVENT                    CDCl3
NS                         50000
DS                             4
SWH                    32679.738 Hz
FIDRES                 0.498653 Hz
AQ                     1.0027508 sec
RG                         32768
DW                        15.300 usec
DE                         7.10 usec
TE                        303.0 K
D1                     1.00000000 sec
d11                    0.03000000 sec
DELTA                  0.89999998 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                         5.00 usec
PL1                       -3.00 dB
SFO1                125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                     98.00 usec
PL2                        3.00 dB
PL12                      23.00 dB
PL13                      32.00 dB
SFO2                500.1320005 MHz

F1 - Acquisition parameters
ND0                            1
TD                           128
SFO1                 500.132 MHz
FIDRES               7.812500 Hz
SW                       1.999 ppm
FnMODE                      QF

F2 - Processing parameters
SI                        262144
SF                  125.7577921 MHz
WDW                         EM
SSB                          0
LB                        0.50 Hz
GB                           0
PC                        1.40

F1 - Processing parameters
SI                          1024
MC2                         QF
SF                  500.1300000 MHz
WDW                       SINE
SSB                          0
LB                        0.30 Hz
GB                         0.1
```



5c

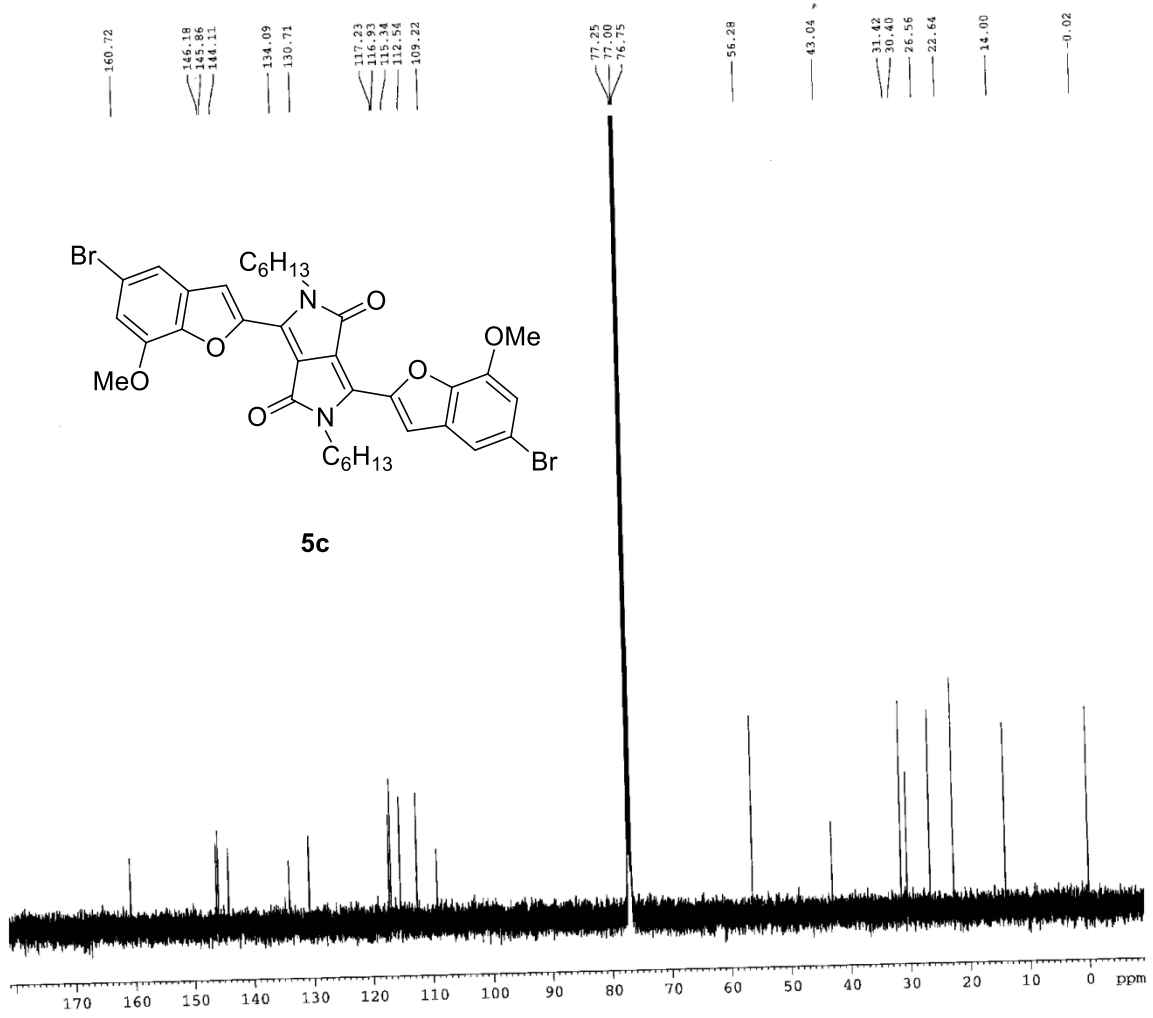
BRUKER

Current Data Parameters
NAME AP252-nowy
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160222
Time 12.32
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 912.3
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300246 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



5c

BRUKER

Current Data Parameters
NAME AP252-nowy
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160222
Time 13.54
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 35600
DS 4
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027508 sec
RG 32768
DW 15.300 usec
DE 7.10 usec
TE 303.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

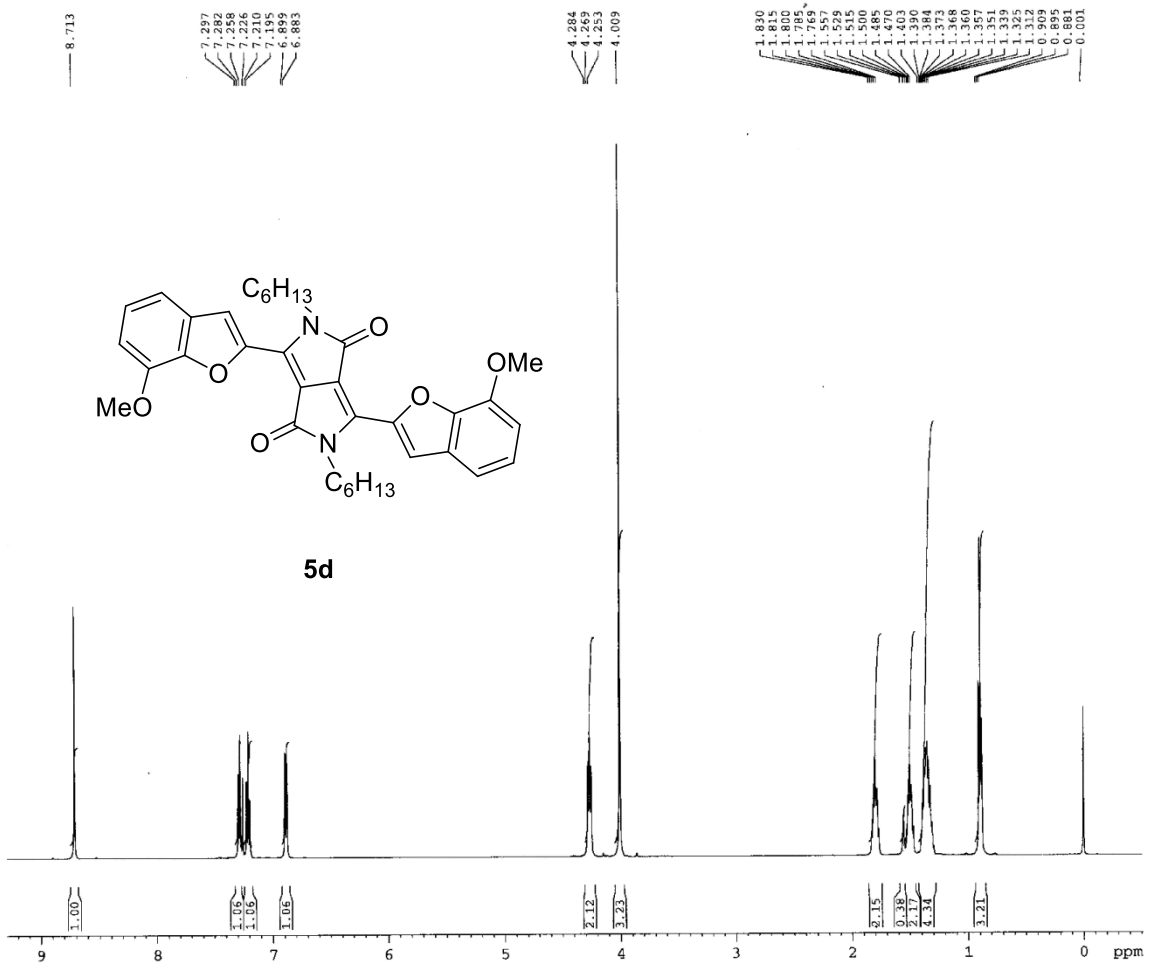
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -3.00 dB
SFO1 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.00 usec
PL2 3.00 dB
PL12 23.00 dB
PL13 32.00 dB
SFO2 500.1320005 MHz

F1 - Acquisition parameters
ND0 1
TD 128
SFO1 500.132 MHz
FIDRES 7.812500 Hz
SW 1.999 ppm
FnMODE QF

F2 - Processing parameters
SI 262144
SF 125.7577917 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0.30 Hz
GB 0.1



5d

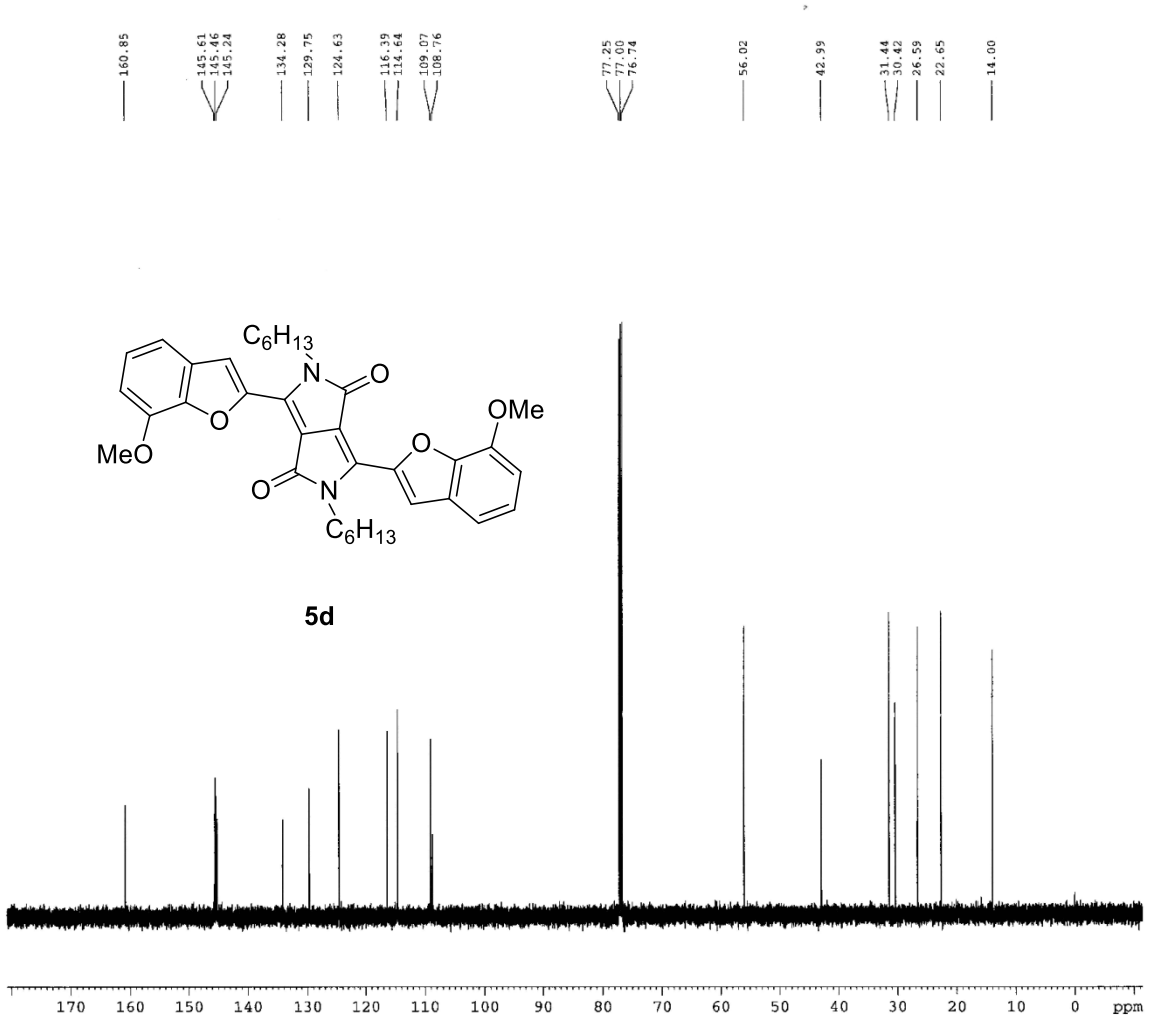
BRUKER

Current Data Parameters
NAME BK-1228
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160113
Time 10.32
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 128
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 2.50 usec
PL1 0.00 dB
SFO1 500.1325006 MHz

F2 - Processing parameters
SI 32768
SF 500.1300246 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



BRUKER

Current Data Parameters
NAME BK-1228
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160113
Time 10.49
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1170
DS 4
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027508 sec
RG 32768
DW 15.300 usec
DE 7.10 usec
TE 303.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

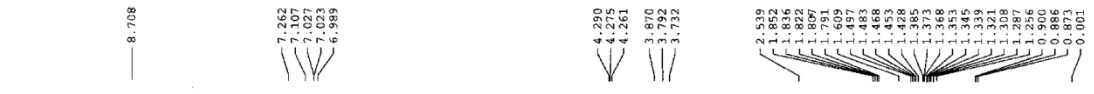
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -3.00 dB
SFO1 125.7703643 MHz

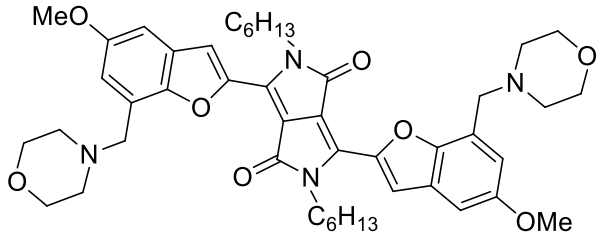
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.00 usec
PL2 3.00 dB
PL12 23.00 dB
PL13 32.00 dB
SFO2 500.1320005 MHz

F1 - Acquisition parameters
ND0 1
TD 128
SFO1 500.132 MHz
FIDRES 7.812500 Hz
SW 1.999 ppm
FnMODE QF

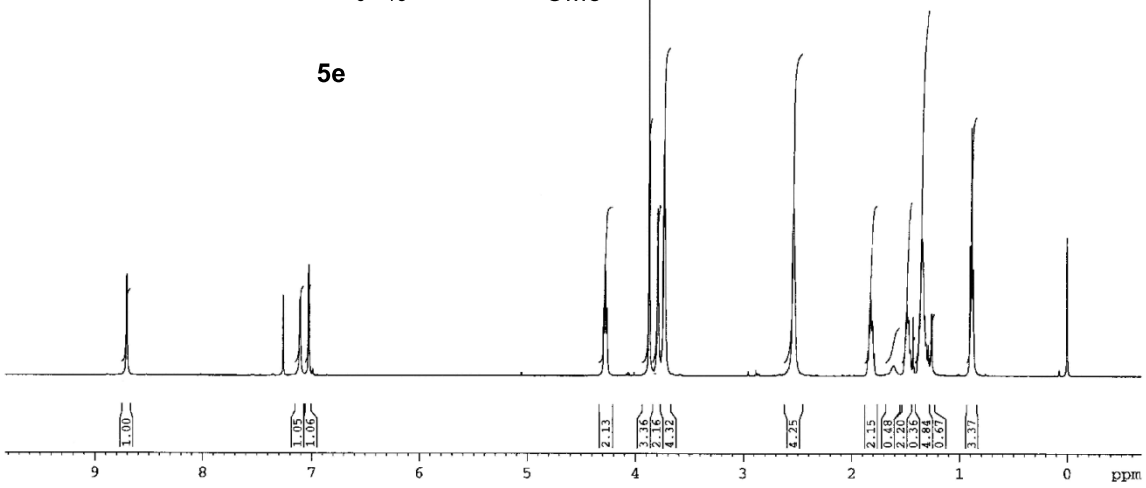
F2 - Processing parameters
SI 262144
SF 125.7577935 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0.30 Hz
GB 0.1

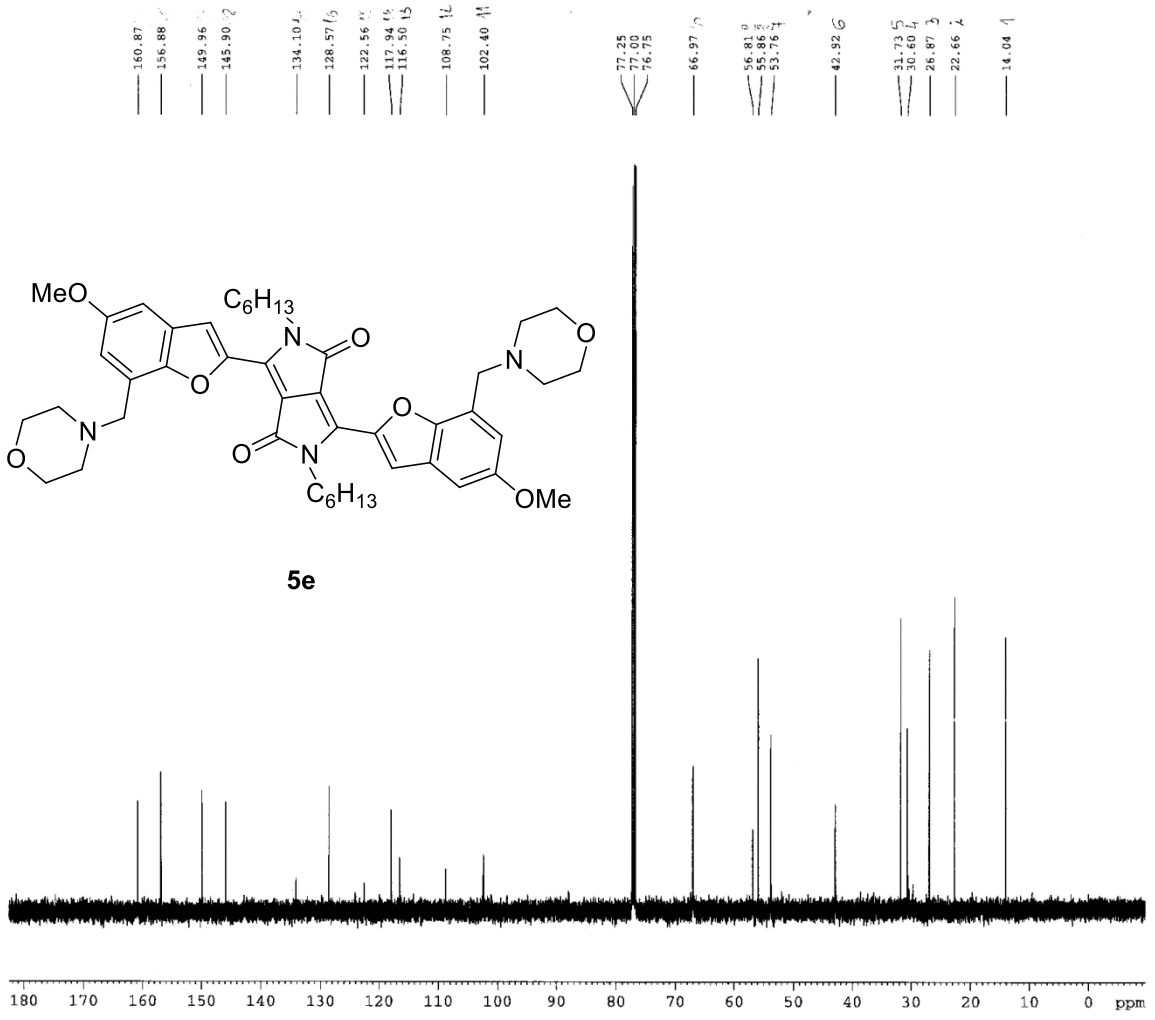




5e

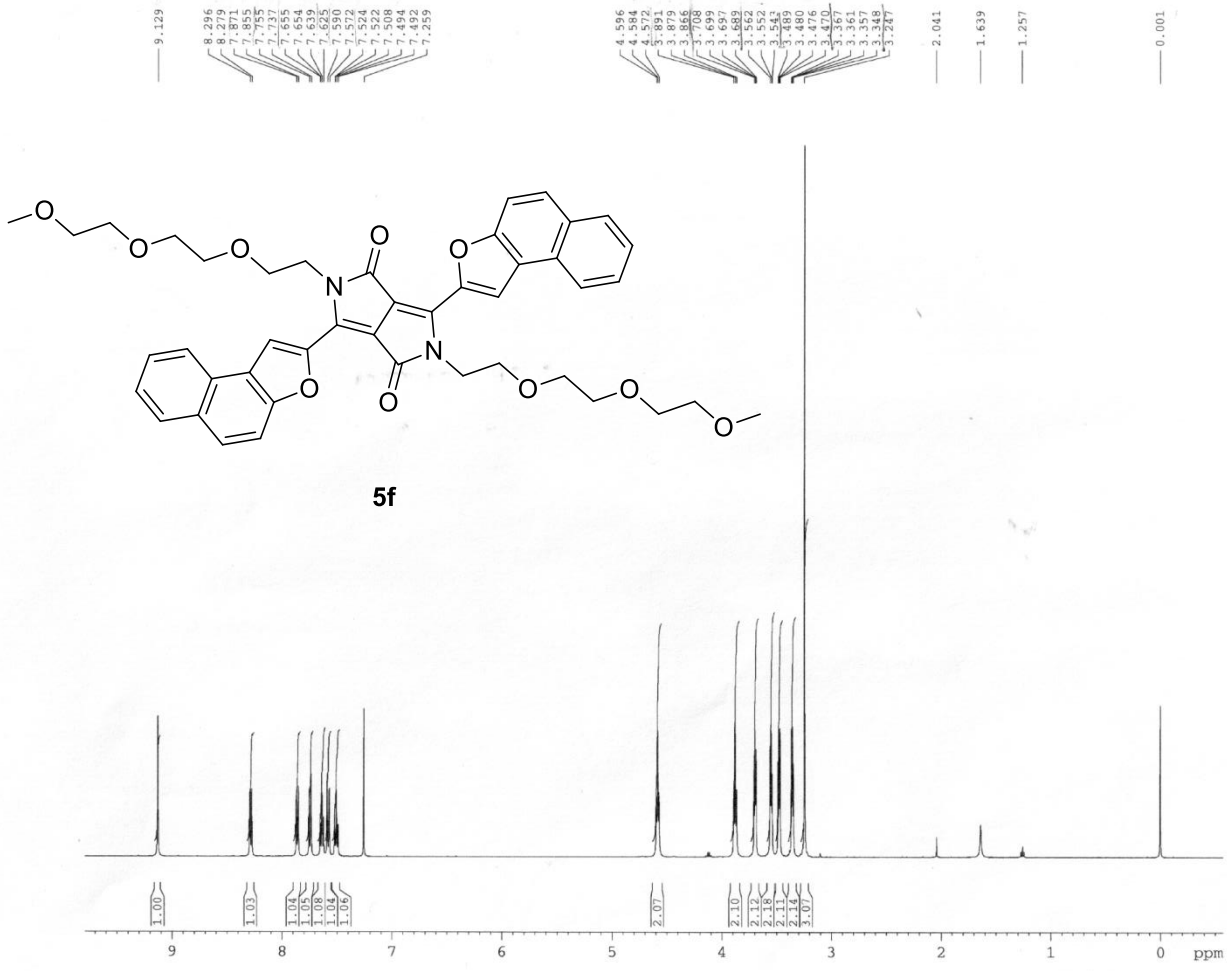



```
Current Data Parameters
NAME                  BK-1229
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20160115
Time                   15.06
INSTRUM                  DRX
PROBHD    5 mm TBI 1H/13
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       32
DS                        0
SWH               10330.578 Hz
FIDRES            0.157632 Hz
AQ                3.1719923 sec
RG                    143.7
DW                   48.400 usec
DE                    6.78 usec
TE                   303.0 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                     1H
P1                    8.20 usec
PL1                   5.00 dB
SFO1          500.1330885 MHz

F2 - Processing parameters
SI                       32768
SF            500.1300225 MHz
WDW                         no
SSB                          0
LB                    0.00 Hz
GB                           0
PC                    1.00
```



BRUKER

```
Current Data Parameters
NAME                  BK-1229
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20160115
Time                  15.42
INSTRUM                 DRX
PROBHD   5 mm TBI 1H/13
PULPROG                zgpg
TD                    65536
SOLVENT               CDCl3
NS                     1515
DS                        4
SWH               32679.738 Hz
FIDRES            0.498653 Hz
AQ                1.0027508 sec
RG                    32768
DW                   15.300 usec
DE                    7.10 usec
TE                   303.0 K
D1                1.00000000 sec
d11               0.03000000 sec
DELTA             0.89999998 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                    13C
P1                     5.00 usec
PL1                   -3.00 dB
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 98.00 usec
PL2                    3.00 dB
PL12                  23.00 dB
PL13                  32.00 dB
SFO2           500.1320005 MHz

F1 - Acquisition parameters
ND0                         1
TD                        128
SFO1               500.132 MHz
FIDRES            7.812500 Hz
SW                    1.999 ppm
FnMODE                   QF

F2 - Processing parameters
SI                     262144
SF             125.7577934 MHz
WDW                      EM
SSB                       0
LB                     0.50 Hz
GB                        0
PC                     1.40

F1 - Processing parameters
SI                       1024
MC2                      QF
SF             500.1300000 MHz
WDW                    SINE
SSB                       0
LB                     0.30 Hz
GB                      0.1
```



5f

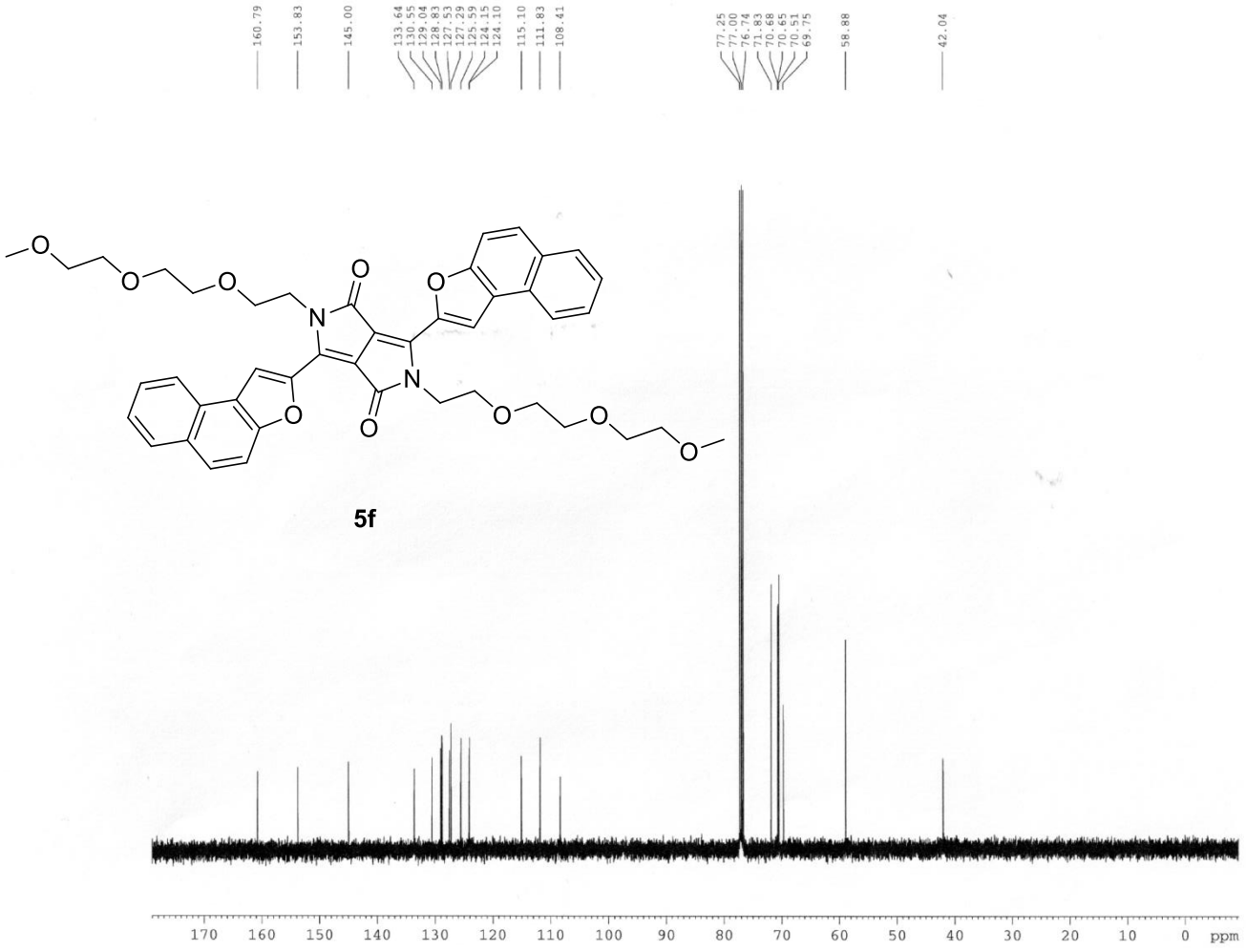
BRUKER

Current Data Parameters
NAME XY87
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150424
Time 13.56
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 161.3
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 2.50 usec
PL1 0.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300245 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



BRUKER

Current Data Parameters
NAME XY87
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150424
Time 14.11
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1500
DS 4
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027508 sec
RG 32768
DW 15.300 usec
DE 7.10 usec
TE 303.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

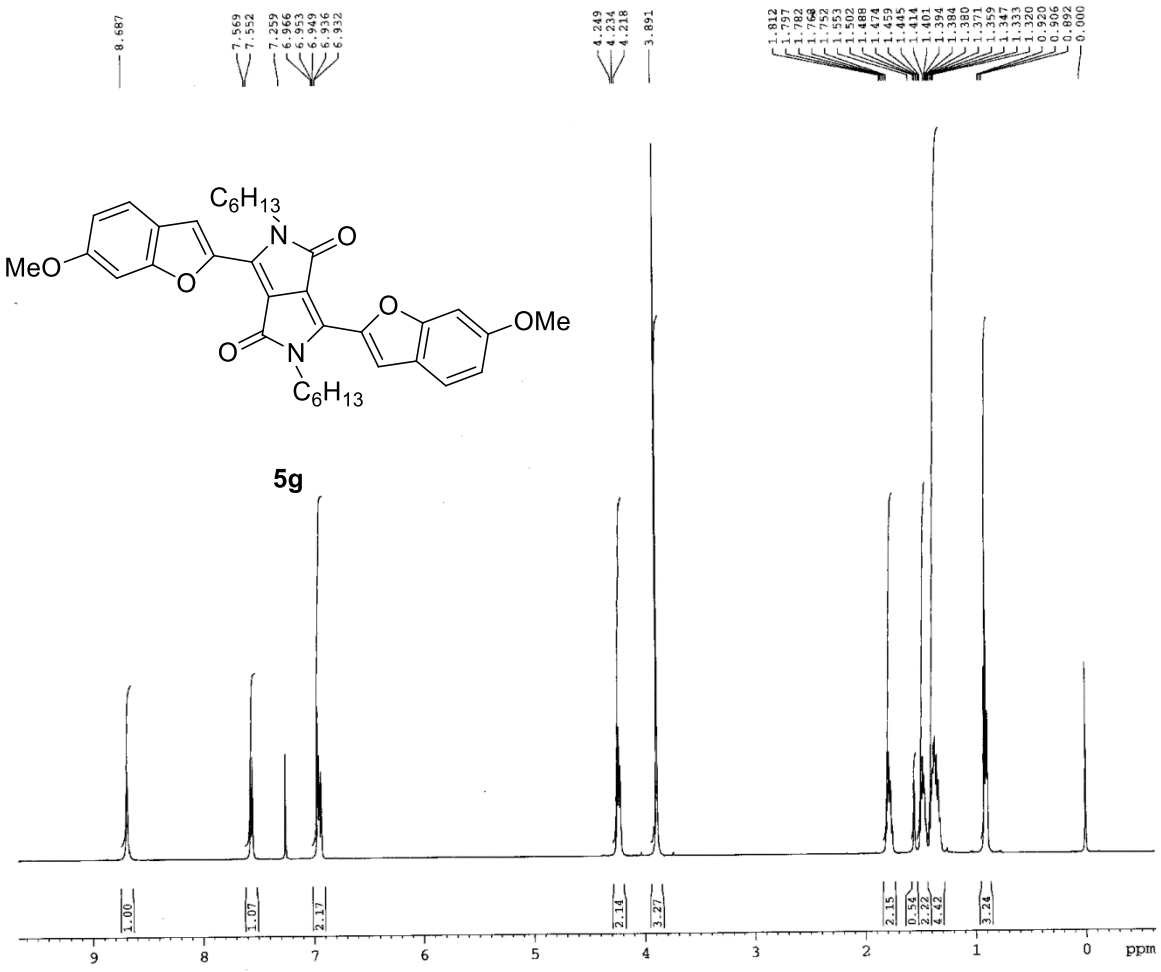
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -3.00 dB
SFO1 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.00 usec
PL2 3.00 dB
PL12 23.00 dB
PL13 32.00 dB
SFO2 500.1320005 MHz

F1 - Acquisition parameters
ND0 1
TD 128
SFO1 500.132 MHz
FIDRES 7.812500 Hz
SW 1.999 ppm
FnMODE QF

F2 - Processing parameters
SI 262144
SF 125.7577939 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0.30 Hz
GB 0.1



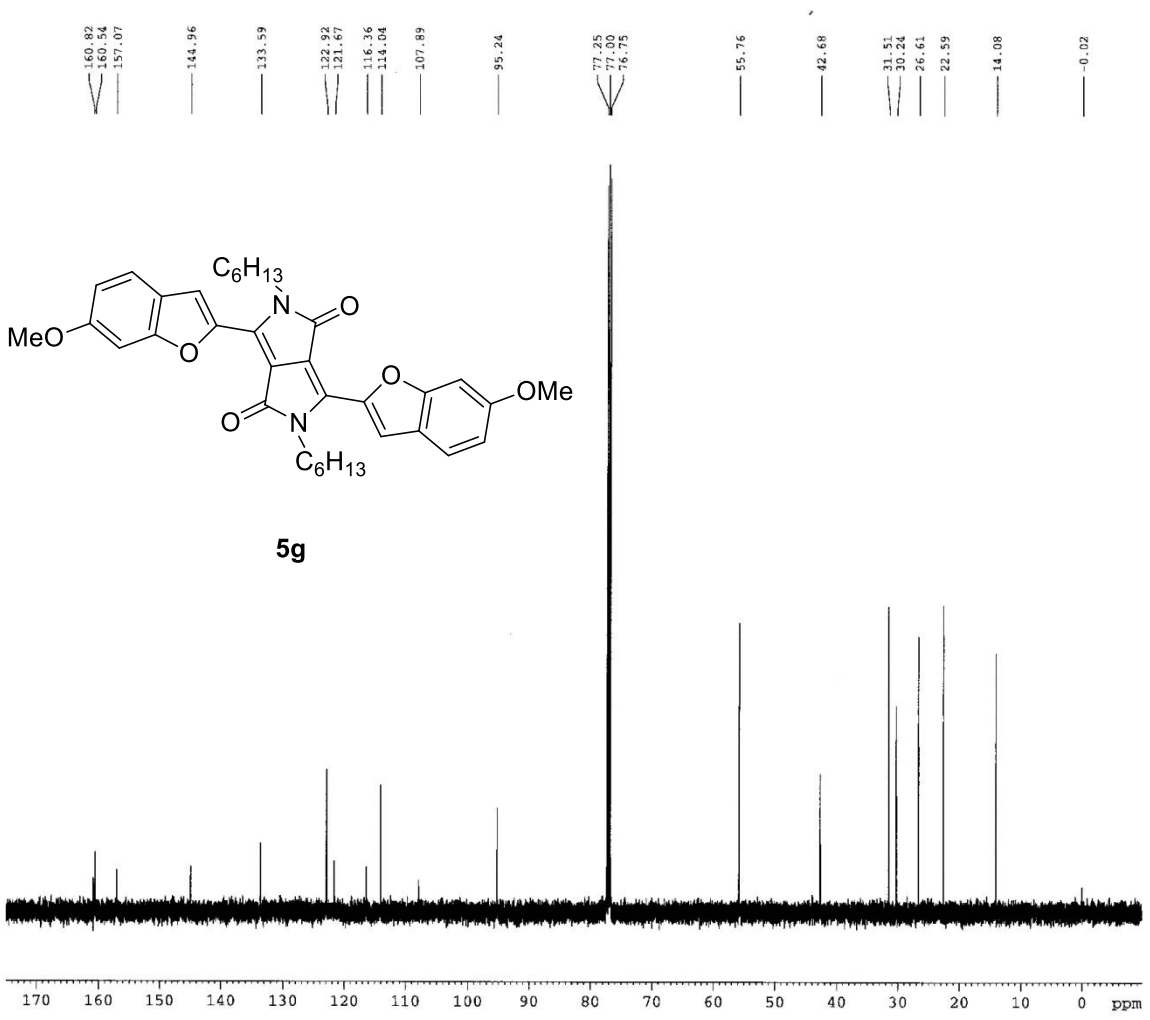
BRUKER

Current Data Parameters
NAME BK-1182-new
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160115
Time 13.37
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 228.1
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300237 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



BRUKER

Current Data Parameters
NAME BK-1182-new
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160115
Time 14.08
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1650
DS 4
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027508 sec
RG 32768
DW 15.300 usec
DE 7.10 usec
TE 303.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -3.00 dB
SFO1 125.7703643 MHz

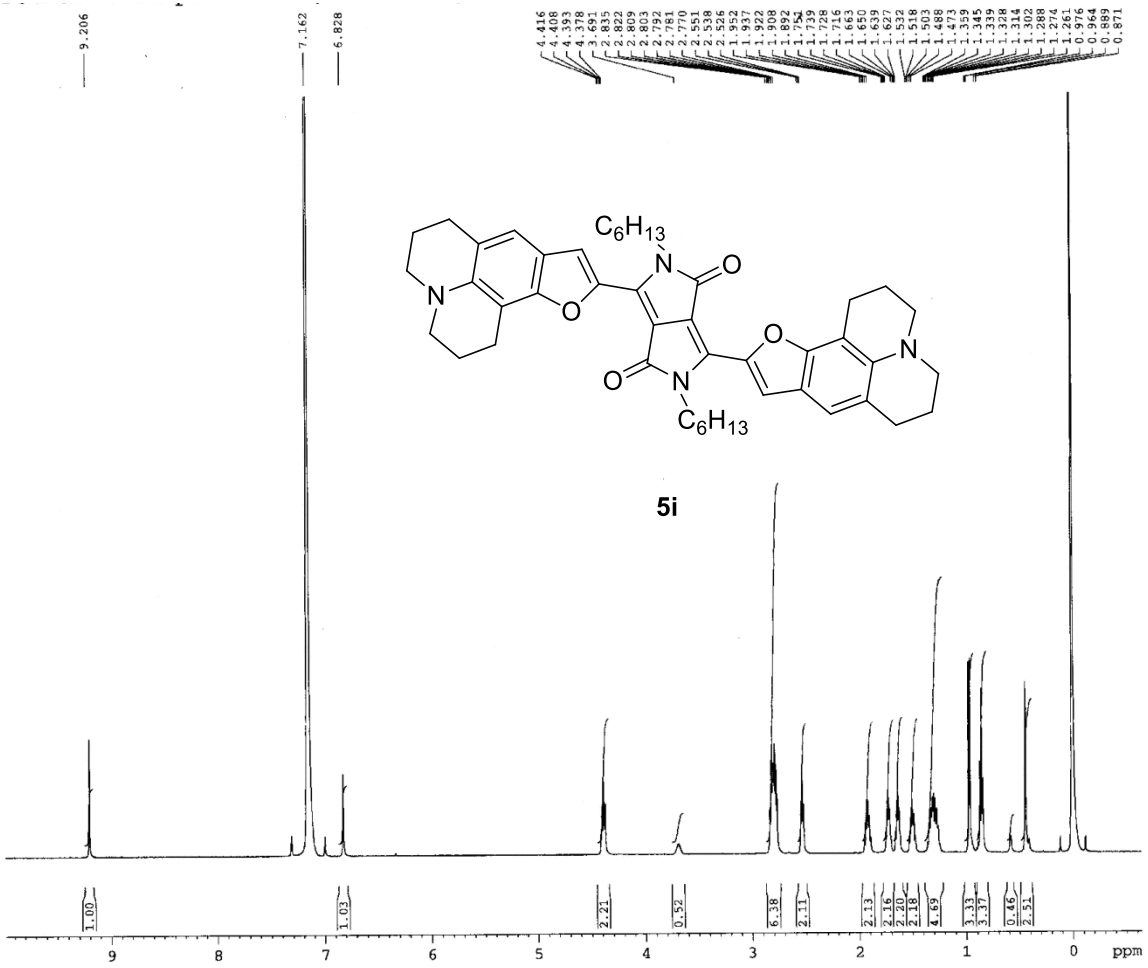
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.00 usec
PL2 3.00 dB
PL12 23.00 dB
PL13 32.00 dB
SFO2 500.1320005 MHz

F1 - Acquisition parameters
ND0 1
TD 128
SFO1 500.132 MHz
FIDRES 7.812500 Hz
SW 1.999 ppm
FnMODE QF

F2 - Processing parameters
SI 262144
SF 125.7577927 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

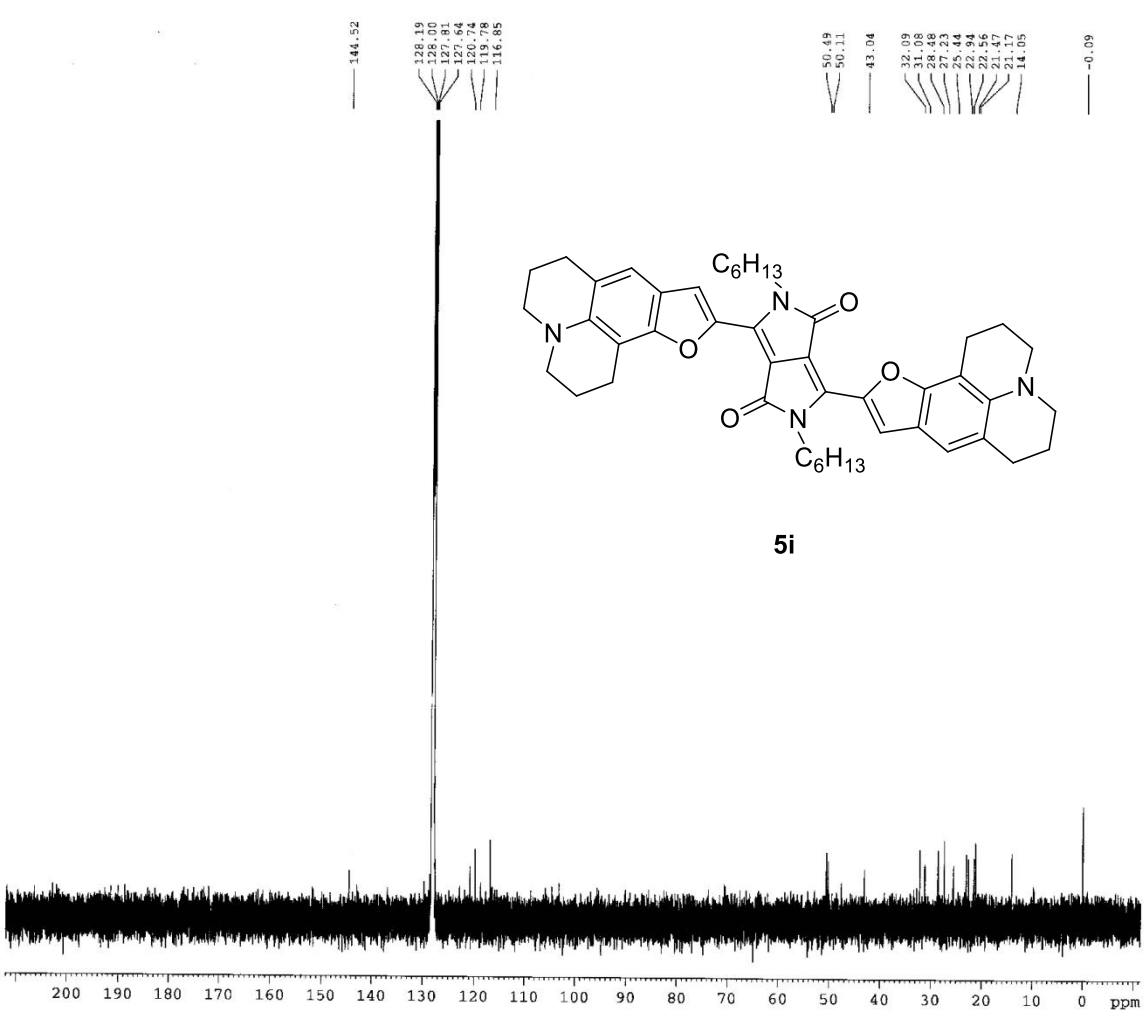
F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0.30 Hz
GB 0.1

13C



5i

```
Current Data Parameters
NAME                  AP257-C6H6
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20160211
Time                       13.13
INSTRUM                      DRX
PROBHD    5 mm TBI 1H/13
PULPROG                     zg30
TD                         65536
SOLVENT                    C6D6
NS                          128
DS                            0
SWH                   10330.578 Hz
FIDRES                0.157632 Hz
AQ                    3.1719923 sec
RG                        574.7
DW                       48.400 usec
DE                        6.78 usec
TE                       343.0 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                         8.20 usec
PL1                        5.00 dB
SFO1                500.1330885 MHz

F2 - Processing parameters
SI                         32768
SF                  500.1300552 MHz
WDW                           EM
SSB                            0
LB                          0.50 Hz
GB                             0
PC                          1.00
```



5i

BRUKER

Current Data Parameters
NAME AP257-C6D6
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160226
Time 10.30
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zgpg
TD 65536
SOLVENT C6D6
NS 10000
DS 4
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027508 sec
RG 32768
DW 15.300 usec
DE 7.10 usec
TE 343.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

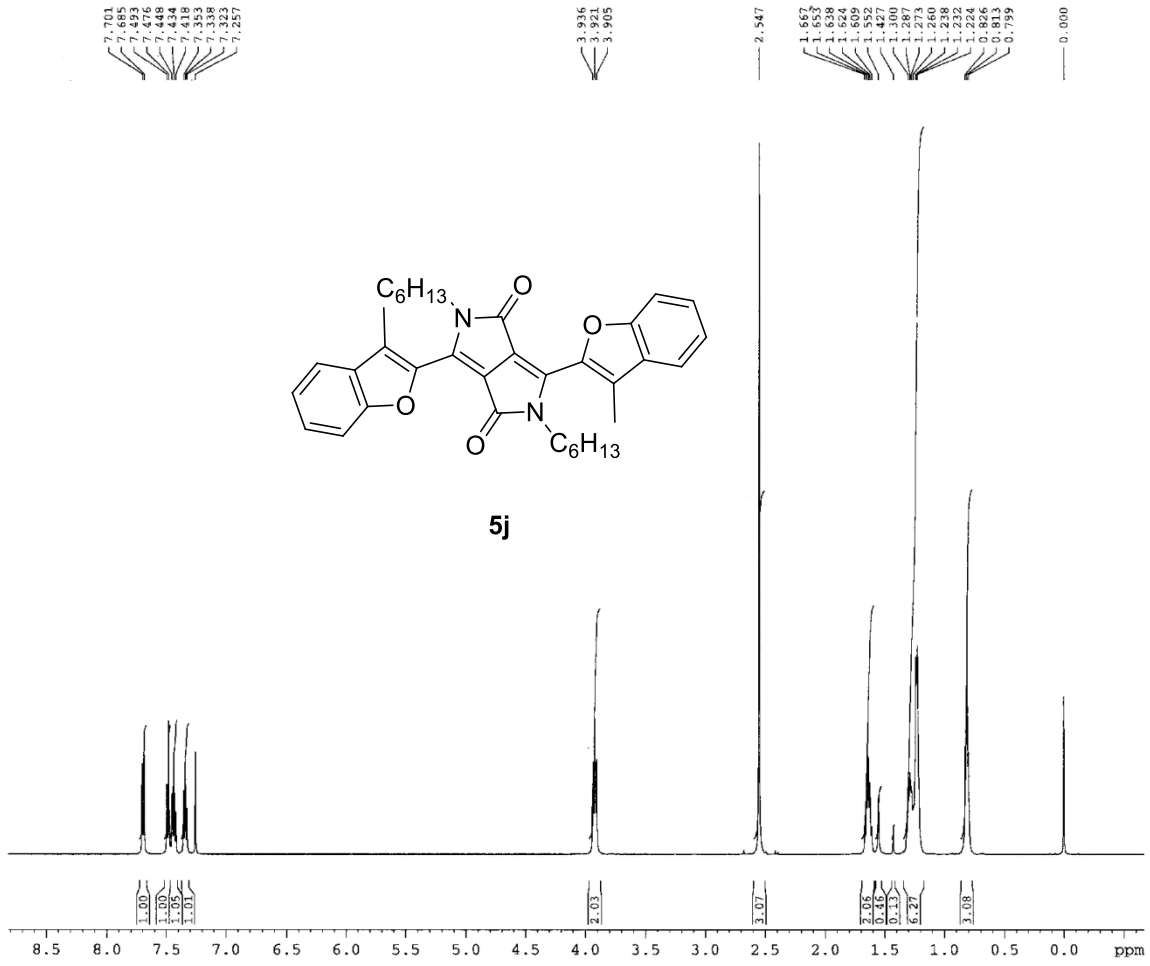
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -3.00 dB
SFO1 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.00 usec
PL2 3.00 dB
PL12 23.00 dB
PL13 32.00 dB
SFO2 500.1320005 MHz

F1 - Acquisition parameters
ND0 1
TD 128
SFO1 500.132 MHz
FIDRES 7.812500 Hz
SW 1.999 ppm
FnMODE QF

F2 - Processing parameters
SI 262144
SF 125.7577490 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0.30 Hz
GB 0.1



5j

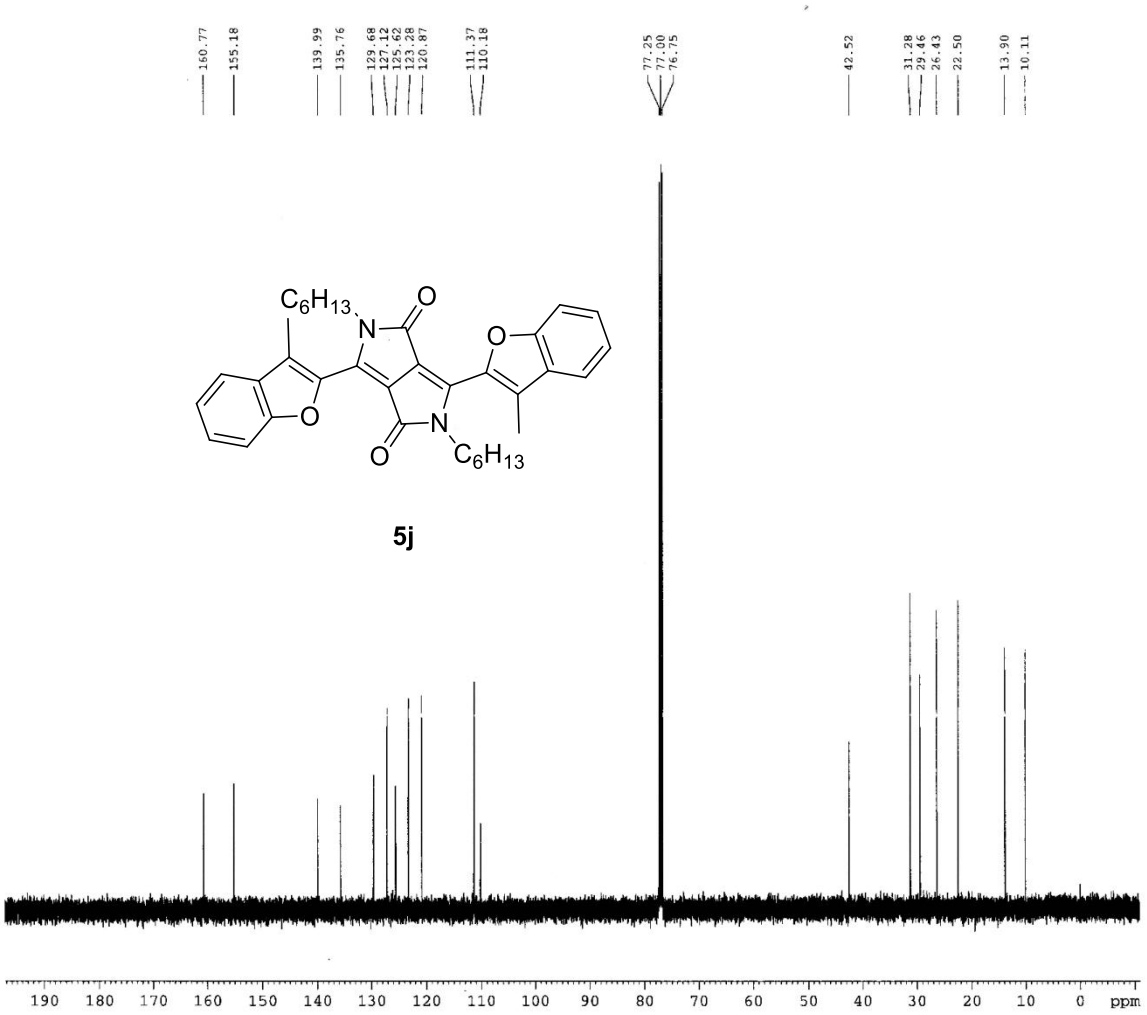
BRUKER

Current Data Parameters
NAME AP237
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151119
Time 16.18
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 228.1
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300254 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



5j

BRUKER

Current Data Parameters
NAME AP237
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151119
Time 16.40
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1149
DS 4
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027508 sec
RG 32768
DW 15.300 usec
DE 7.10 usec
TE 303.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

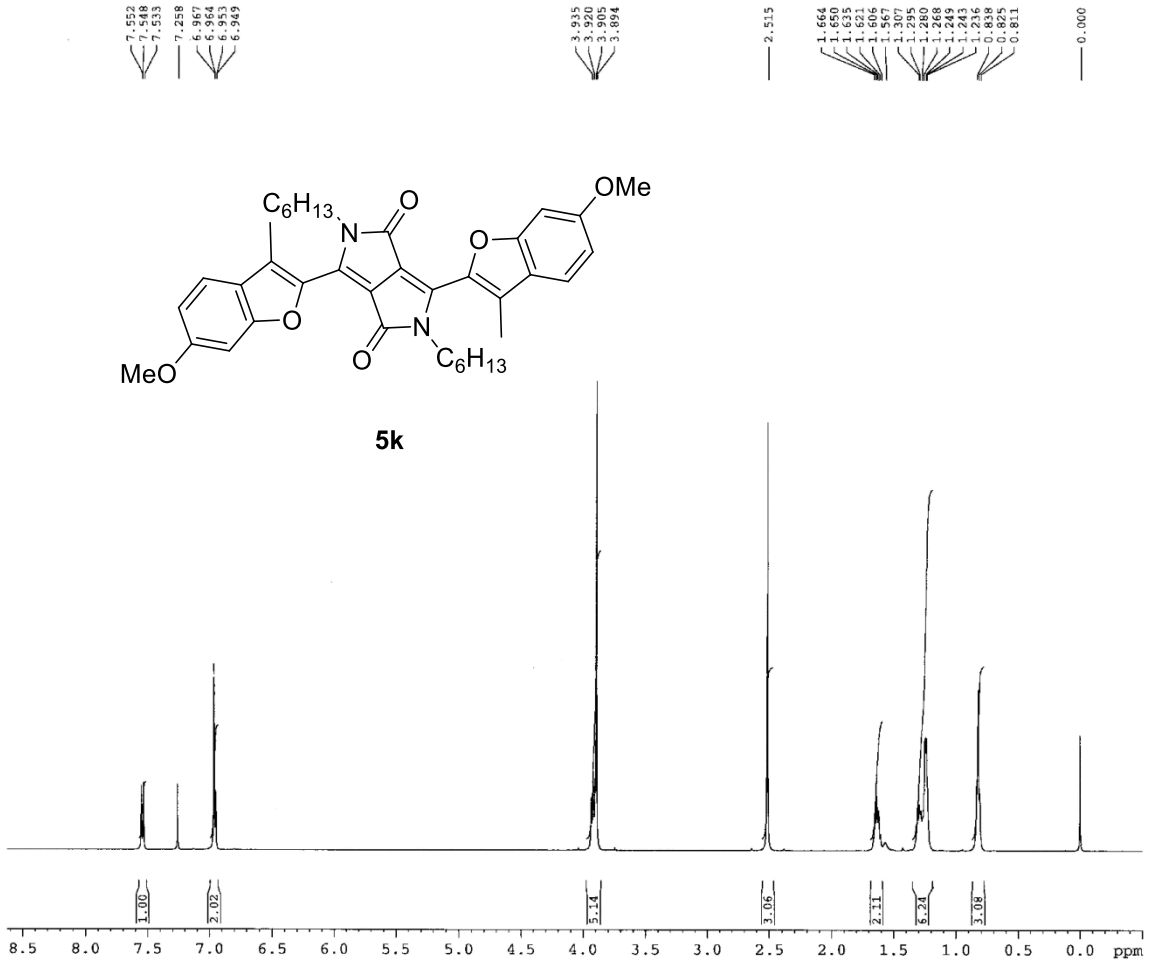
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -3.00 dB
SFO1 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.00 usec
PL2 3.00 dB
PL12 23.00 dB
PL13 32.00 dB
SFO2 500.1320005 MHz

F1 - Acquisition parameters
ND0 1
TD 128
SFO1 500.132 MHz
FIDRES 7.812500 Hz
SW 1.999 ppm
FnMODE QF

F2 - Processing parameters
SI 262144
SF 125.7577931 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0.30 Hz
GB 0.1



5k

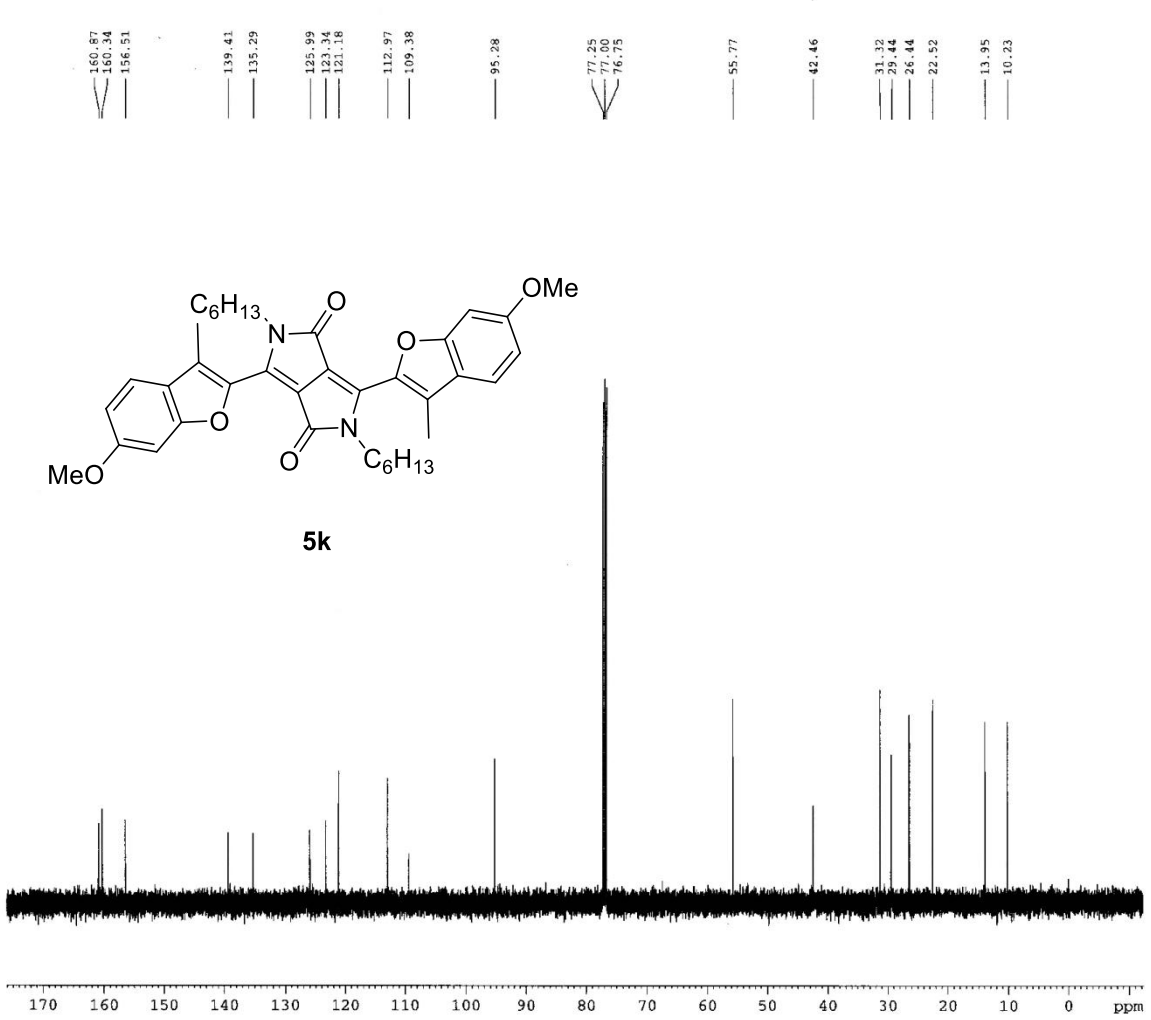
BRUKER

Current Data Parameters
NAME AP250
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160114
Time 15.26
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 203.2
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 500.1330885 MHz

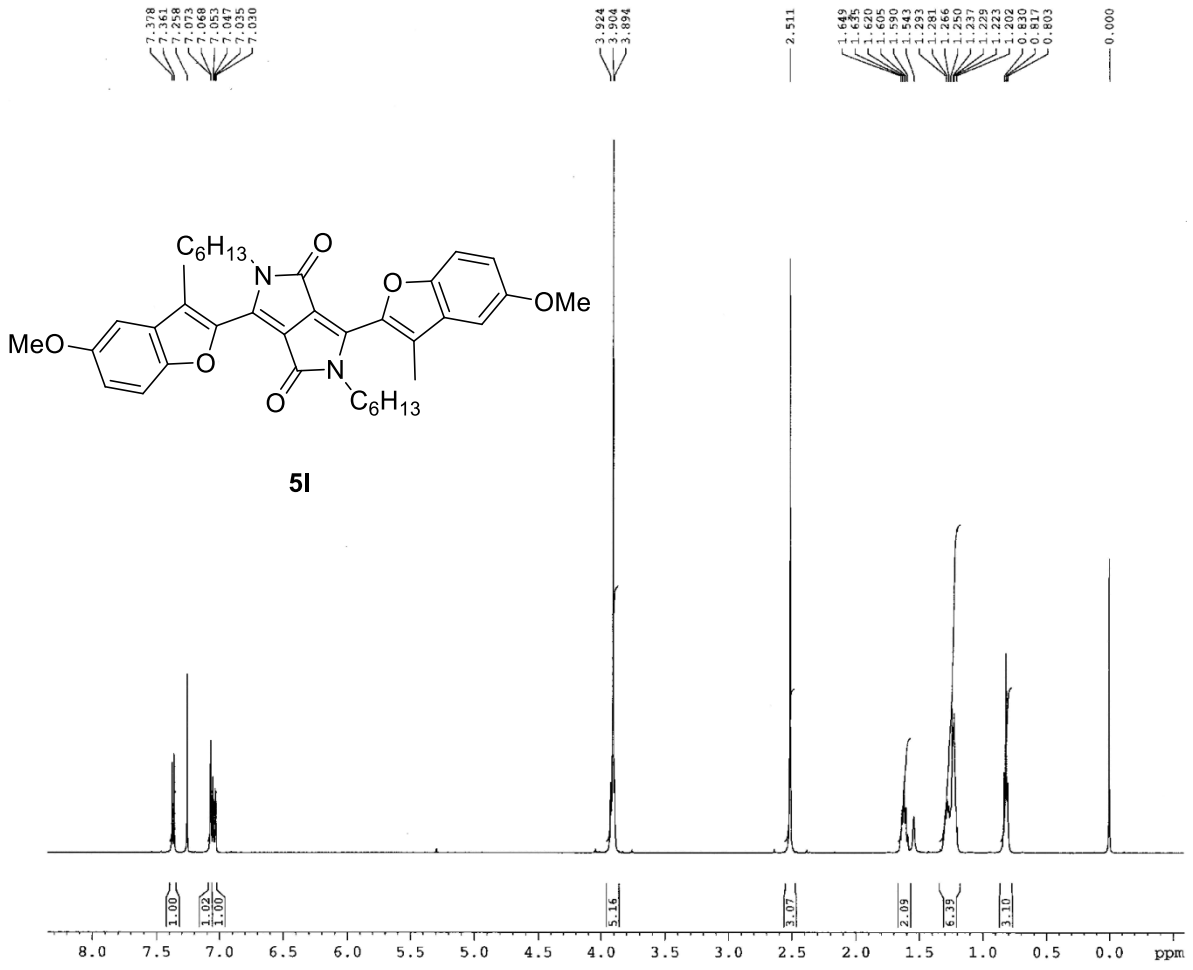
F2 - Processing parameters
SI 32768
SF 500.1300240 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



5k

BRUKER

```
Current Data Parameters
NAME                  AP250
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20160114
Time                  15.49
INSTRUM                 DRX
PROBHD   5 mm TBI 1H/13
PULPROG                zgpg
TD                    65536
SOLVENT               CDCl3
NS                      540
DS                        4
SWH               32679.738 Hz
FIDRES            0.498653 Hz
AQ                1.0027508 sec
RG                    32768
DW                   15.300 usec
DE                    7.10 usec
TE                   303.0 K
D1                1.00000000 sec
d11               0.03000000 sec
DELTA             0.89999998 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                     5.00 usec
PL1                   -3.00 dB
SFO1            125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 98.00 usec
PL2                    3.00 dB
PL12                  23.00 dB
PL13                  32.00 dB
SFO2            500.1320005 MHz

F1 - Acquisition parameters
ND0                       1
TD                      128
SFO1              500.132 MHz
FIDRES            7.812500 Hz
SW                    1.999 ppm
FnMODE                   QF

F2 - Processing parameters
SI                   262144
SF              125.7577928 MHz
WDW                      EM
SSB                       0
LB                     0.50 Hz
GB                        0
PC                     1.40

F1 - Processing parameters
SI                     1024
MC2                      QF
SF              500.1300000 MHz
WDW                    SINE
SSB                       0
LB                     0.30 Hz
GB                      0.1
```



5l

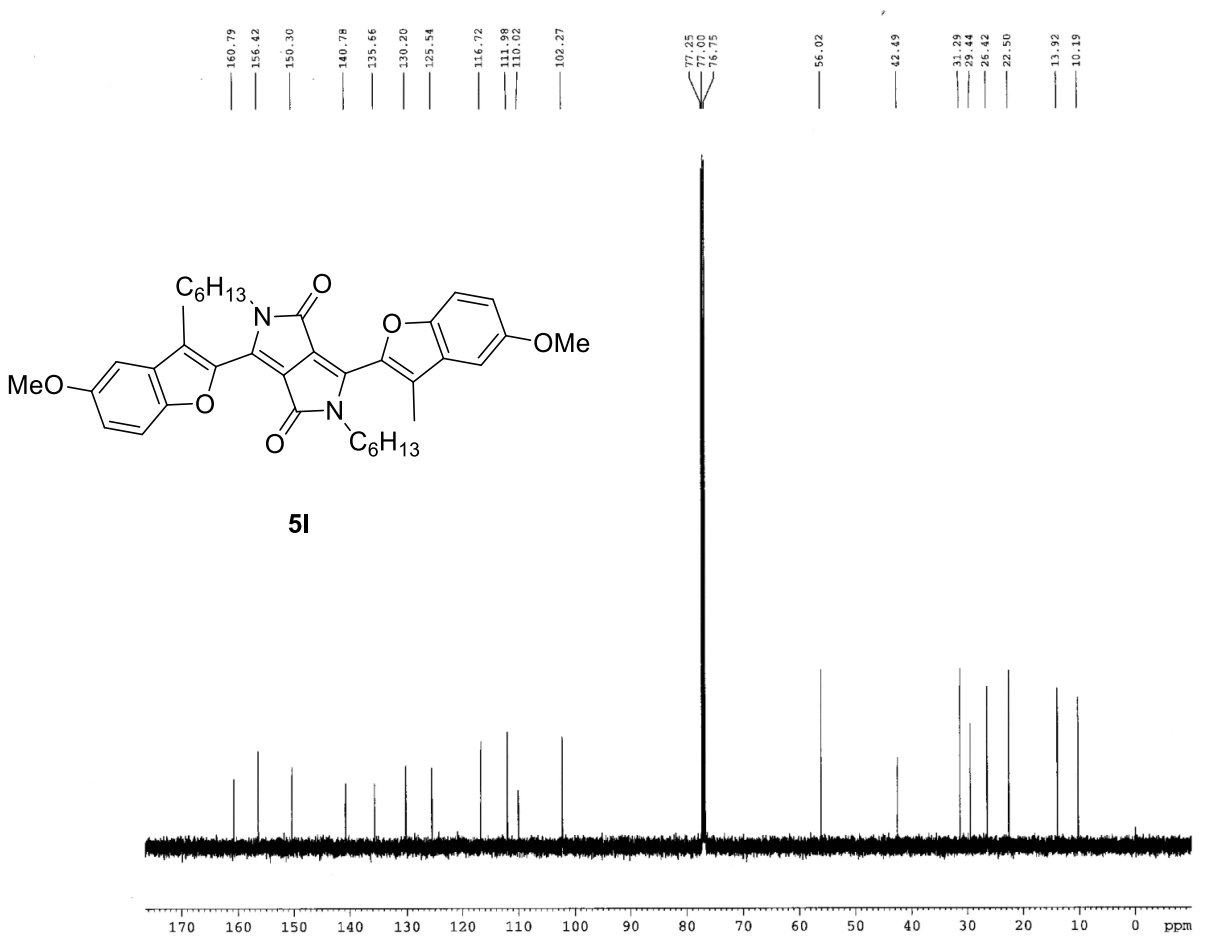
BRUKER

Current Data Parameters
NAME AP263
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160121
Time 11.39
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 322.5
DW 48.400 usec
DE 6.78 usec
TE 303.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 5.00 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300246 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



5l

BRUKER

Current Data Parameters
NAME AP263
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160121
Time 12.34
INSTRUM DRX
PROBHD 5 mm TBI 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 1999
DS 4
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027508 sec
RG 32768
DW 15.300 usec
DE 7.10 usec
TE 303.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -3.00 dB
SFO1 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 98.00 usec
PL2 3.00 dB
PL12 23.00 dB
PL13 32.00 dB
SFO2 500.1320005 MHz

F1 - Acquisition parameters
ND0 1
TD 128
SFO1 500.132 MHz
FIDRES 7.812500 Hz
SW 1.999 ppm
FnMODE QF

F2 - Processing parameters
SI 262144
SF 125.7577925 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 500.1300000 MHz
WDW SINE
SSB 0
LB 0.30 Hz
GB 0.1

[1] S. Vedachalam, J. Zeng, B.K. Gorityala, M. Antonio and X.-W. Liu, *Org.Lett*., 2010, **12**, 352-355.
[2] T. Horaguchi, C. Tsukada, E. Hasegawa, T. Shimizu, T. Suzuki and K. Tanemura, *J. Heterocyclic Chem.*, 1991, **28**, 1261-1272.
[3] W. Zhou, W. Chena and L. Wang, *Org. Biomol. Chem*., 2012, **10**, 4172-4178.
[4] Z. Fu, Z. Li, Y. Song, R. Yang, Y. Liu and H. Cai, *J. Org. Chem.*, 2016, **81**, 2794-2803.